

# **ADVANCED ACCELERATED TESTING METHODOLOGY FOR LONG-TERM LIFE PREDICTION OF POLYMER COMPOSITES**

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## **SUMMARY**

The advanced accelerated testing methodology (Advanced ATM) for the long-term life prediction of polymer composites exposed to an actual loading having general stress and temperature history is proposed in this paper. First, four conditions as the basis of Advanced ATM are introduced with the scientific bases. One of these conditions is the concept of viscoelastic compliance to describe the effect of load and temperature history. Second, the formulations of creep compliance and time-temperature shift factors of matrix resin are carried out. The creep compliance of matrix resin performs an important role for time and temperature dependence of long-term life of polymer composites. And the formulations of long-term life of polymer composites under an actual loading are carried out based on the four conditions. Third, the applicability of Advanced ATM is confirmed by comparing the predicted life to the experimental ones in the case of cyclic loading where loading and unloading are repeated to CFRP laminates.

*Key words: Polymer composites, CFRP, Life prediction, Viscoelasticity*

## **INTRODUCTION**

Carbon fiber reinforced plastics (CFRP) are now being used for the primary structures of airplanes, spacecrafts and others as well as ships, in which the high reliability should be kept during the long-term operation. Therefore, it would be expected that the accelerated testing methodology for the long-term life prediction of composite structures exposed under the actual environments of temperature, water, and others must be established.

A strategy of accelerated testing is shown as the following steps, 1) Data collections of accelerated testing, 2) Durability Design, 3) Development of highly reliable structures. First, the accelerated testing methodology should be established for polymer composites. Our developed methodology will be generic and can be applied to centrifuge, generator, flywheel, aircraft, wind turbine, marine and automobile.

In this paper, we propose an advanced accelerated testing methodology (Advanced ATM) which can be applied to the life prediction of polymer composites exposed to an actual load and environment history. First, four conditions as the basis of Advanced ATM are introduced with the scientific bases. One of these conditions is the concept

of viscoelastic compliance to describe the effect of load and temperature history. Second, the formulations of creep compliance and time-temperature shift factors of matrix resin are carried out. The creep compliance of matrix resin performs an important role for time and temperature dependence of long-term life of polymer composites. And the formulations of long-term life of polymer composites under an actual loading are carried out based on the four conditions. Third, the applicability of Advanced ATM is confirmed by comparing the predicted life to the experimental ones in the case of cyclic loading where loading and unloading are repeated to CFRP laminates.

### **ADVANCED ATM**

The Advanced ATM is established with four following conditions: (A) the same time-temperature superposition principle (TTSP) is applicable for both non-destructive viscoelastic behavior and destructive strength properties of matrix resin and their composites; (B) strength variation is caused by viscoelastic compliance of matrix resin; (C) failure probability is independent of time, temperature, frequency and stress ratio; (D) strength degradation is caused by linear cumulative damage of cyclic loading.

A key component of the Advanced ATM is also the empirical observation (A), which has been demonstrated its applicability for various polymeric composite materials and their structures [1]. Based on the condition (A), the master curves of constant strain rate (CSR), creep and fatigue strengths in the wide range of time and temperature can be determined by the data measured by these tests under the short-term and elevated temperature conditions. With the condition (B), it is possible to calculate the strength variation by the viscoelastic compliance of matrix resin determined by the creep compliance of matrix resin and the history of load and temperature changed with time. With the condition (C), the reference strength and the failure probability can be obtained by measuring CSR strength at an arbitrary strain rate under room temperature. With the condition (D), it is possible to calculate the strength degradation by load cycles undergoing to the linear cumulative damage law. The formulation for long-term fatigue strength of polymer composites exposed to an actual load and environment history are conducted under the four conditions of Advanced ATM.

The procedure for determining the materials parameters in the formulation of the Advanced ATM is illustrated in Figure 1. First, the change in modulus or compliance of viscoelastic matrix resin is measured over time at a constant temperature. The tests are repeated for several elevated temperatures, which results in several modulus or compliance curves with the function of time. The time-temperature shift factor are then determined by shifting the viscoelastic modulus or compliance curves at the several temperatures into time scale to form a master curve of the modulus or compliance at a reference temperature. The time-temperature shift factor is thus the measure of the acceleration of the life of matrix resin by means of the elevated temperatures. The next step is to obtain the creep strength master curves. This step consists of two parts. The first part is to determine CSR strength master curve of the composites from the CSR loading tests conducted at a single strain rate and several elevated temperatures using the time-temperature shift factor for matrix resin, and the second part is to convert CSR strength master curve to the creep strength master curve of the composites. Third, the master curves of fatigue strength of the composites at

zero stress ratio are determined by conducting the fatigue tests at several stress levels, a single frequency, stress ratio (zero stress ratio) and temperature using CSR strength master curve. Finally, the master curves of fatigue strength at an arbitrary stress ratio are determined by the creep strength master curve and the fatigue strength master curves at zero stress ratio. Through these procedures, all of materials parameters in the formulations can be determined for the long-term fatigue strength at an arbitrary load condition in which the stress and temperature arbitrarily changed with time.

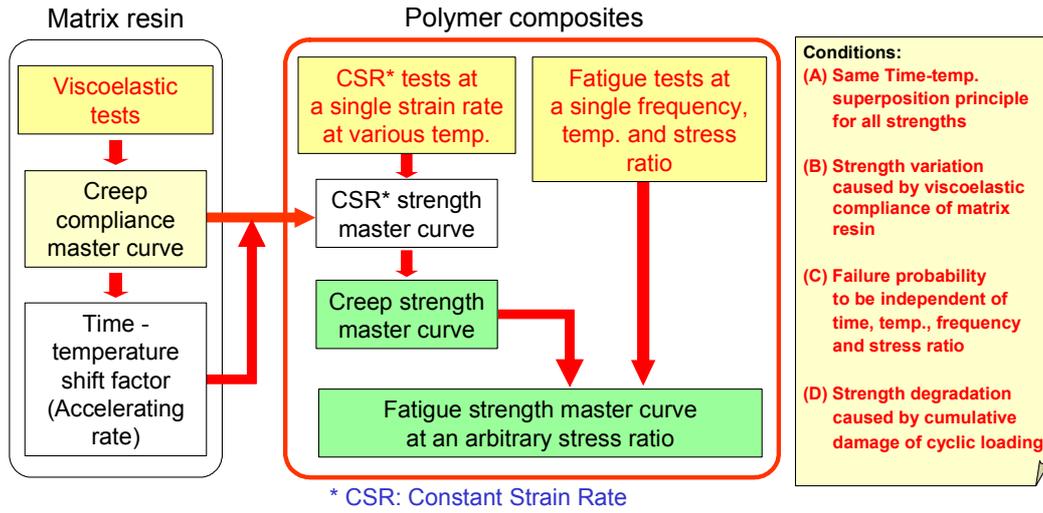


Figure 1 Procedure of Advanced ATM for polymer composites

## FORMULATION

### Long-term Fatigue Strength of Polymer Composites under Actual Loading

The long-term fatigue strength  $\sigma_f(t', T_0)$  under actual loading can be formulated based on the four conditions of Advanced ATM.

$$\log \sigma_f(t', P_f, N_f, T_0) = \log \sigma_s(t'_0, T_0) + \frac{1}{\alpha} \log [-\ln(1 - P_f)] - n_r \log \left[ \frac{D^*(t', T_0)}{D_c(t'_0, T_0)} \right] - n_f \log \left[ \frac{N_f}{N_0} / (1 - k_D) \right] \quad (1)$$

where the first term  $\sigma_s(t'_0, T_0)$  is the scale parameter of CSR strength measured at an initial reduced time  $t'_0$  at a reference temperature  $T_0$  which is the static strength at glassy state.

The second term shows Weibull distribution of failure probability  $P_f$  in which the shape parameter is  $\alpha$ . The  $\alpha$  is independent of time to failure, temperature, frequency and stress ratio from the condition (C) based on Christensen's theory [2].

The third term shows the variation caused by the viscoelastic compliance  $D^*$  of matrix resin defined by the following equation based on the condition (B). The  $n_r$  in this term is the parameter to be dependent on the failure mechanism.

$$D^*(t', T_0) = \frac{\varepsilon(t', T_0)}{\sigma(t', T_0)} = \frac{\int_0^{t'} D_c(t' - \tau', T_0) \frac{d\sigma(\tau')}{d\tau'} d\tau'}{\sigma(t', T_0)}, \quad (2)$$

where  $D_c$  is the creep compliance of matrix resin.  $t'$  is the reduced time at the reference temperature  $T_0$  which is shown by the following equation based on the condition (A).

$$t' = \int_0^t \frac{d\tau}{a_{T_0}(T(\tau))}, \quad (3)$$

where  $a_{T_0}$  is the time-temperature shift factor for the creep compliance of matrix resin.

The fourth term shows the degradation caused by cumulative damage of cyclic loading. In this term,  $N_f$  is the number of cycles to failure at the final step.  $N_0$  is the reference number of cycles to failure = 1/2 and  $n_f$  is the parameter to be dependent on the failure mechanism.  $k_D$  is defined as the following equation based on the condition (D).

$$k_D = \sum_{i=1}^n \frac{n_i}{N_{fi}} < 1, \quad (4)$$

where  $n_i$  and  $N_{fi}$  are the number of cycles and the number of cycles to failure at the loading of step  $i$ , respectively.

### Creep Compliance of Matrix Resin

The formulation for the master curve of creep compliance of matrix resin  $D_c$  in Equation (1) and the time-temperature shift factor of matrix resin  $a_{T_0}(T)$  in Equation (3) should be performed for the long-term fatigue strength of polymer composites under actual loading. The master curve of  $D_c$  can be represented by two tangential lines, whose slopes are  $m_g$  and  $m_r$ , respectively. With these parameters, the master curve of  $D_c$  can be fit with the following equation,

$$\log D_c = \log D_c(t'_0, T_0) + \log \left[ \left( \frac{t'}{t'_0} \right)^{m_g} + \left( \frac{t'}{t'_g} \right)^{m_r} \right], \quad (5)$$

where  $t'_0$  is an initial reduced time at a reference temperature  $T_0$ .  $t'_g$  is the reduced glassy time at a reference temperature  $T_0$ .  $m_g$  and  $m_a$  are the parameters.

Alternatively, the viscoelastic behaviors of the matrix resin can be represented by the storage modulus  $E'$  which can easily be measured with experimental devices such as the dynamic mechanical analyzer (DMA) conducted at various frequencies and temperatures. Note that  $D_c$  can approximately be obtained from  $E'$  by using

$$D_c = 1/E' \quad (6)$$

### CSR, Creep and Fatigue Strength of Polymer Composites

For the CSR strength  $\sigma_s$ , Equation (1) can be simplified to following equation,

$$\log \sigma_s(t', T_0) = \log \sigma_s(t'_0, T_0) + \frac{1}{\alpha} \log[-\ln(1 - P_f)] - n_r \log \left[ \frac{D_c(t'/2, T_0)}{D_c(t'_0, T_0)} \right] \quad (7)$$

For the creep strength  $\sigma_c$ , Equation (1) can be simplified to following equation,

$$\log \sigma_c(t', T_0) = \log \sigma_s(t'_0, T_0) + \frac{1}{\alpha} \log[-\ln(1 - P_f)] - n_r \log \left[ \frac{D_c(t', T_0)}{D_c(t'_0, T_0)} \right], \quad (8)$$

For the fatigue strength  $\sigma_f$ , Equation (1) can be simplified to following equations,

$$\log \sigma_f(t', T_0) = \log \sigma_s(t'_0, T_0) + \frac{1}{\alpha} \log[-\ln(1 - P_f)] - n_r \log \left[ \frac{D^*(t', T_0)}{D_c(t'_0, T_0)} \right] - n_f \log[2N_f], \quad (9)$$

where

$$D^*(t', T_0) = \frac{1}{2} D_c(t', T_0) + \frac{1}{2} D_c \left( \frac{1}{4f'}, T_0 \right) \quad (10)$$

### Cyclic Creep and Fatigue Strength of Polymer Composites

The following equation for cyclic creep and fatigue strengths under the cyclic loadings shown by Figure 2 are obtained from Equation (1).

$$\log \sigma_f(t', P_f, N_f, T_0) = \log \sigma_s(t'_0, T_0) + \frac{1}{\alpha} \log[-\ln(1 - P_f)] - n_r \log \left[ \frac{D^*(t', T_0)}{D_c(t'_0, T_0)} \right] - n_{f1} \log(2N_{f1}) - n_{f2} \log(2N_{f2}) \quad (11)$$

where the parameters for the cyclic creep loading are shown as follows,

$$D^*(t', T_0) = D_c[t', T_0] + \sum_{i=1}^{n-1} [D_c[t'+(n-i)(t'_1+t'_u), T_0] - D_c[t'+(n-i)(t'_1+t'_u) - t'_1, T_0]], \quad n_{f1}=0$$

and the parameters for the cyclic fatigue loading are shown as follows

$$D^*(t', T_0) = \frac{1}{2} D_c[t', T_0] + \frac{1}{2} D_c \left[ \frac{1}{4f'}, T_0 \right] + \frac{1}{2} \sum_{i=1}^{n-1} [D_c[t'+(n-i)(t'_1+t'_u), T_0] - D_c[t'+(n-i)(t'_1+t'_u) - t'_1, T_0]]$$

$N_{f1}$  and  $N_{f2}$  in Equation (11) are the number of cycles to failure under the cyclic loadings corresponding to the frequency  $f_1$  and  $f_2$  shown by the red curve in the right graph of Figure 2 and the green curves in both graphs of this figure.

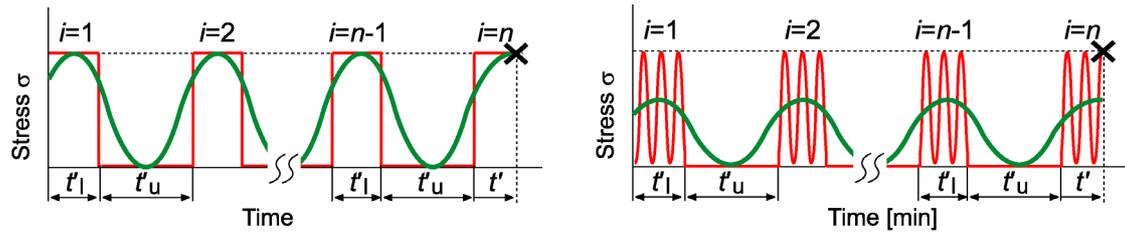


Figure 2 Cyclic creep and fatigue loadings

## EXPERIMENTS

### Preparation of Specimens

CFRP laminates employed is T800S/3900-2B quasi-isotropic laminate, which consists of unidirectional T800S carbon fiber and 3900 epoxy resin with toughened interlayer by polymer particles for the use as the wing structures of aircraft. The stacking sequence of this CFRP laminates is  $[90]_8$  and  $[45/0/-45/90]_{2s}$ . The CFRP laminates is cured at  $180^\circ\text{C}$  for 2 hours and then post cured at  $170^\circ\text{C}$  for 10 hours. The volume fraction of fibers in the CFRP laminate was approximately 60%, and the thickness was approximately 3.0 mm.

### Experimental Procedure

First, the storage modulus of CFRP laminates of T800S/3900-2B  $[90]_8$  against various frequencies at various temperatures were measured using DMA testing machine and the master curve of creep compliance of matrix resin was determined. Second, three point bending tests to CFRP laminates of T800S/3900-2B  $[90]_8$  were performed under constant and cyclic creep loads at temperature  $T=45^\circ\text{C}$  and the deflections were measured during loading. Third, three point bending tests to CFRP laminates of T800S/3900-2B  $[45/0/-45/90]_{2s}$  were performed under constant and cyclic creep and fatigue loads at temperature  $T=45^\circ\text{C}$  and the time to failure were measured. The configuration of specimen of three point bending test for T800S/3900-2B  $[45/0/-45/90]_{2s}$  are shown in Figure 4.

Material		T800S/3900-2B	
Material			
Stacking sequence		$[90]_8$	$[45/0/-45/90]_{2s}$

Figure 3 CFRP laminates

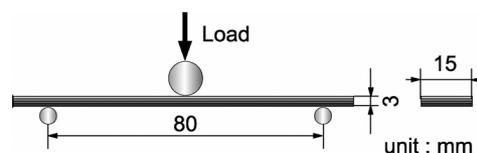


Figure 4 Testing method and specimen configuration for three point bending tests

## RESULTS AND DISCUSSION

### Master Curve of Creep Compliance for Matrix Resin

The storage modulus of T800S/3900-2B [90]<sub>8</sub> against time of inverse of frequency at various temperatures are shown in the left graph of Figure 5. The smooth master curve of storage modulus can be obtained by shifting horizontally and vertically the measured data as shown by the right graph of Figure 5. The horizontal and vertical parts of time-temperature shift factor are shown in Figure 6.

The master curve of creep compliance of matrix resin can be obtained using the master curve of storage modulus of T800S/3900-2B [90]<sub>8</sub> and the rule of mixture as shown in Figure 7. The formulation for this master curve is performed using Equation (5) and the parameters in this equation are determined as shown on Table 1.

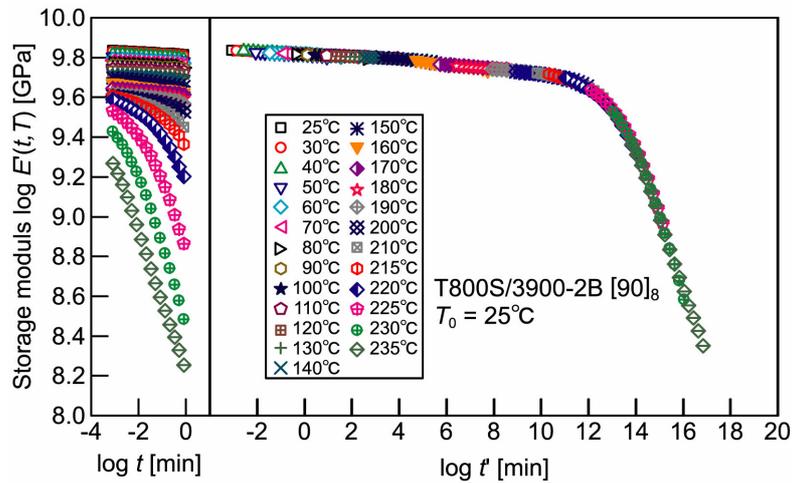


Figure 5 Master curve of storage modulus of T800S/3900-2B [90]<sub>8</sub>

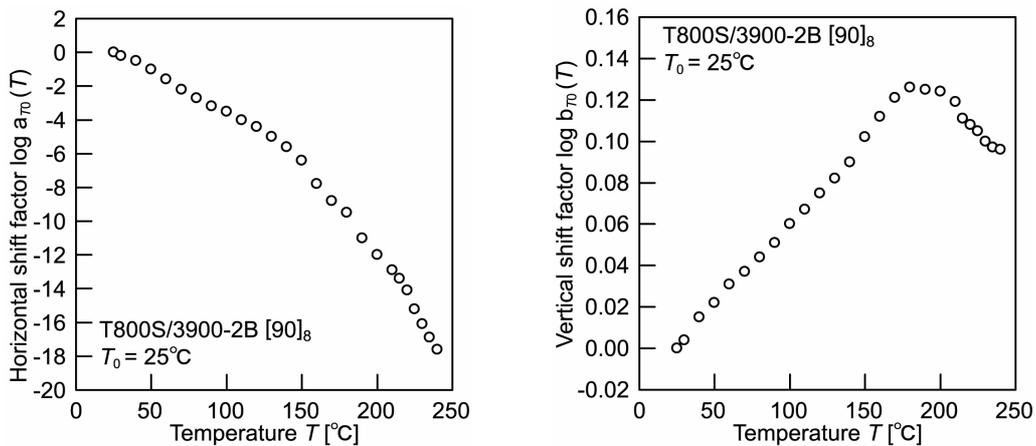


Figure 6 Time-temperature shift factor for storage modulus of T800S/3900-2B [90]<sub>8</sub>

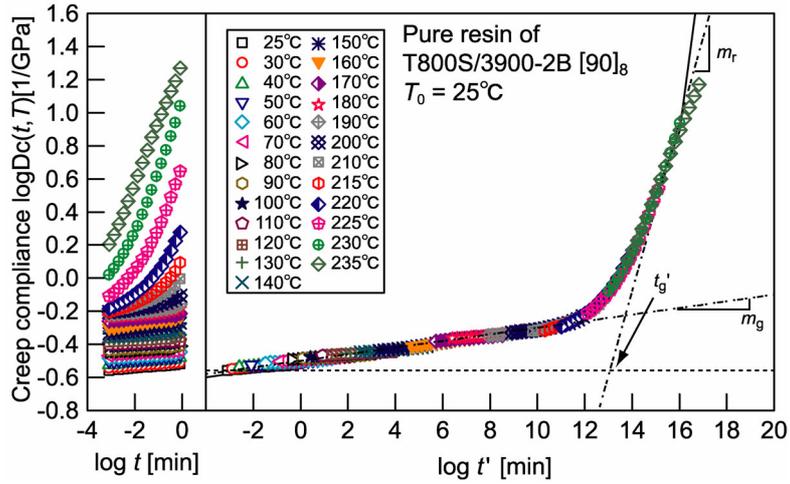


Figure 7 Master curve of creep compliance for matrix resin of T800S/3900-2B

Table 1 Parameters of creep compliance for matrix resin of T800S/3900-2B

$T_0$ [°C]	25
$t'_0$ [min]	1
$D_{c0}(t'_0, T_0)$ [1/GPa]	0.303
$t'_g$ at $T_0$ [min]	$1.60 \times 10^{13}$
$m_g$	0.020
$m_r$	0.490

### Creep Deflections at Constant and Cyclic Creep Loadings

The creep deflections at constant and cyclic creep loadings for T800S/3900-2B [90]<sub>8</sub> are shown in Figure 8. It is clearly shown in this figure that the deflection by cyclic loading is suppressed by the creep recovery during unloading. Therefore, it is presumed that the strength of CFRP laminates under cyclic creep loading is strengthened by the creep recovery of matrix resin during unloading.

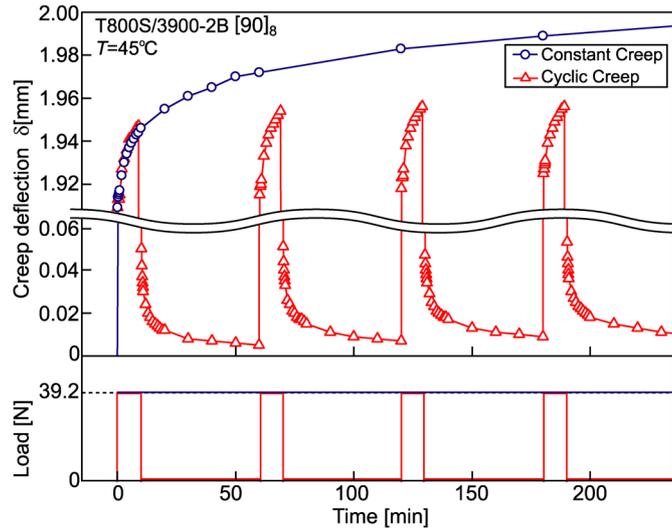


Figure 8 Creep deflections under constant and cyclic creep loadings

### Master Curve of Flexural CSR Strength

The flexural CSR strength of T800S/3900-2B [45/0/-45/90]<sub>2S</sub> against the time to failure at various temperatures are shown in the left graph of Figure 9 and the master curve of this CFRP laminates is obtained using the time-temperature shift factor for the matrix resin as shown in the right side of Figure 9. The solid, dotted, and chain curves show the scale parameter, the failure probability 10% and 90% formulated by Equation (7), respectively.

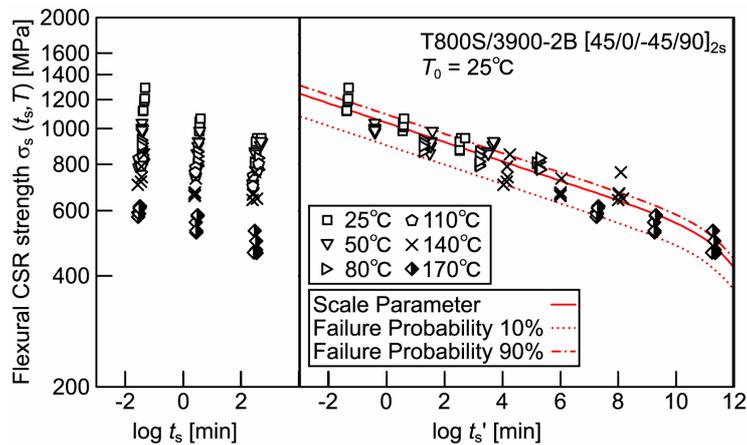


Figure 9 Master curve of flexural CSR strength for T800S/3900-2B [45/0/-45/90]<sub>2S</sub>

### Strength under Constant and Cyclic Creep and Fatigue Loadings

The probability against the time to failure for T800S/3900-2B [45/0/-45/90]<sub>2S</sub> in the cases of constant and cyclic creep and fatigue bending loadings are shown in Figures 10 and 11, respectively. The blue circles and curve in each figure show the experimental data under constant creep and fatigue tests and the formulation by Equations (8) and (9),

respectively. The red circles and curves in each figure show the experimental data under cyclic creep and fatigue tests and the formulation by Equation (11), respectively. The numbers from 2 to 50 in these figures show the number of cycles of loading and unloading. The red curves at  $n_{f2}=0$  in these figures show the case that the damage by the cycles of loading and unloading is negligible. Actually,  $n_{f2}=0.051$  and  $n_{f2}=0.025$  are fitted for both of cyclic loadings, respectively.

Table 2 shows the parameters of formulation shown by Equation (11). The scale parameter  $\sigma_s$  at  $t'_0$  and  $T_0$  is determined by CSR test data and the other parameters are determined by the data by constant and cyclic creep and fatigue tests. The fact that the  $n_{f2}$  for cyclic creep loading is twice of the  $n_{f2}$  for cyclic fatigue is corresponding to the fact that the load amplitude for cyclic creep loading is twice to that for cyclic fatigue loading as shown in Figure 2.

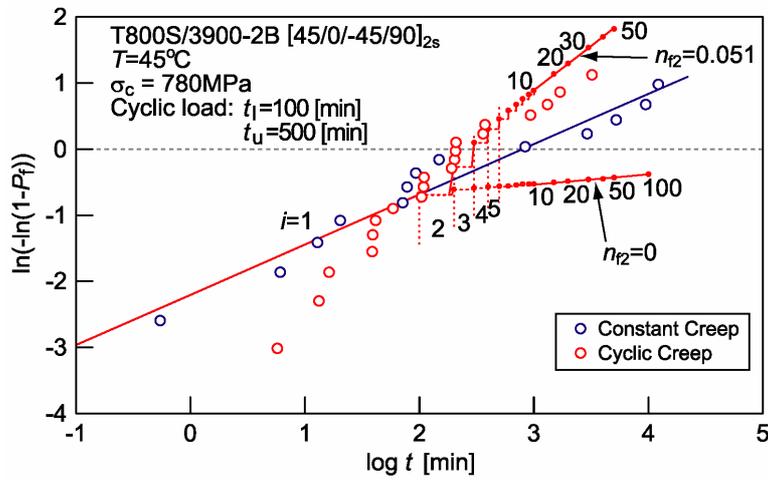


Figure 10 Failure probability against time to failure under constant and cyclic creep loadings

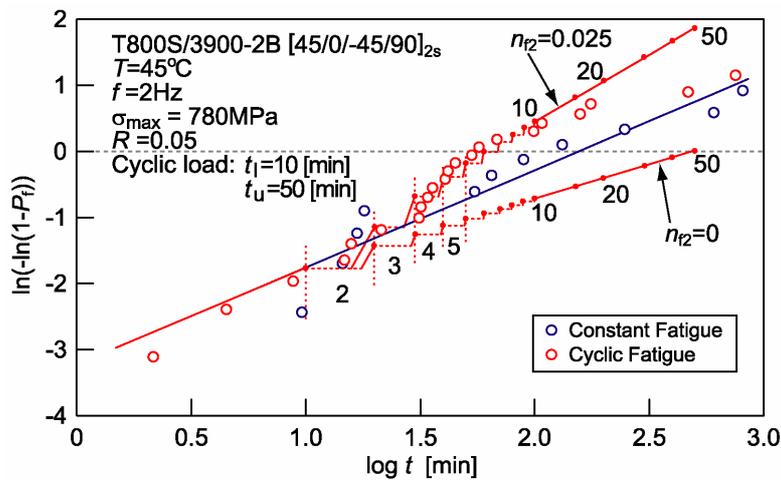


Figure 11 Failure probability against time to failure under constant and cyclic fatigue loadings

Table 2 Parameters of cyclic creep and fatigue flexural strength

	Cyclic creep	Cyclic fatigue
$\sigma_{s,0}$	1037	1037
$\alpha$	9.7	16.0
$n_r$	1.69	1.31
$n_{f1}$	0	0.025
$n_{f2}$	0.051	0.025

### CONCLUSIONS

We proposed an advanced accelerated testing methodology (Advanced ATM) which can be applied to the life prediction of polymer composites exposed to an actual load and environment history. First, four conditions as the basis of Advanced ATM were introduced with the scientific bases. One of these conditions was the concept of viscoelastic compliance to describe the effect of load and temperature history. Second, the formulations of creep compliance and time-temperature shift factors of matrix resin were carried out. And the formulations of long-term life of polymer composites under an actual loading were carried out based on the four conditions. Third, the applicability of Advanced ATM was confirmed by comparing the predicted life to the experimental ones in the case of cyclic loading where loading and unloading are repeated to CFRP laminates.

### REFERENCES

1. Miyano, Y., and Nakada, M., Cai, H., "Formulation of long-term creep and fatigue strengths of polymer composites based on accelerated testing methodology", *Journal of Composite Materials*, Vol.42, pp.1897-1919 (2008).
2. Christensen, R., and Miyano, Y. "Stress intensity controlled kinetic crack growth and stress history dependent life prediction with statistical variability", *Int. Jour. of Fracture*, Vol.137, pp.77-87 (2006).