EVALUATION OF INTERFACIAL STRENGTH IN MICRODROPLET TEST USING EXPERIMENT AND COMPUTATIONAL SIMULATION

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Abstract
Interfacial strength is an important factor that dominates the mechanical properties of composites. This paper presents an evaluation method for fiber/matrix interfacial strength. In this study, we determine interfacial strength by comparing experimental data with FEM computational simulations of the microdroplet tests. We consider first the residual thermal stresses that result upon cooling from the cure temperature to room temperature. Cohesive elements are introduced on the interface of the FE model, and dilatational damage and shear damage, based on continuum damage mechanics, are introduced into the resin. Moreover, it is thought that compressive stress enhances interfacial shear strength. Coulomb's friction law is applied at the debonded interface. We discuss interfacial energy, which is a factor determining mechanical properties. Interfacial strength setting high energy value of traction-separation law might be inappropriate because the separation length becomes longer than the fiber radius, which is probably different from the actual behavior. This study attempts to simulate actual behavior with very low interfacial energy. As a result, it is revealed that the provided interfacial strength by numerical simulation is higher than IFSS (Interfacial Shear Strength).

1. Introduction

Interfacial strength plays a key role in not only static strength [1,2] of the composite material but also the long-term durability [3-5]. Therefore, the authors spent an effort on the measurement of the interfacial strength [6-8]. Interfacial strength of the composite material is measured in the single fiber pull out test [9], the single fiber push in test [10], the single fiber push out test [11], the fiber fragmentation test [12,13], and the microdroplet test [14]. Specimens for microdroplet tests are prepared by attaching an resin droplet to a single fiber and curing it. The specimen is loaded to shear the fiber from the resin droplet by using knife edges and the fiber load is measured then. The microdroplet method was used by several reserchers to evaluate interfacial strength, there are especially many study of interfacial strength evaluation by conbining experimental data with FEM computational simulations of this tests [15,16].

Four following points are to be considerd in numerical simulation of microdroplet test. (1) the accurate thermal residual stresses considered thermal viscoelastic, (2) the damage of the resin when the edge touches the resin droplet, (3) the interfacial debonding used cohesive elements and (4) the transition of interfacial strength by the interfacial compressive stress [17]. Particularly, it is necessary to distinguish contribution of the damage of the resin and the interfacial failure because there are mixed in microdroplet test. Nishikawa et al. [15] simulated the damage of the resin by the damage

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based on continuum mechanics are introduced into resin, but viscoelastic of the resin was not considered. Sockalingam et al. [16] reproduce using FE numerical simulation interfacial debonding as fitting experimental values by interfacial high energy value of traction separation low is estimated. However, this might be inappropriate because separation length becomes longer than fiber radius, which is probably different from the actual behavior.

In this way, there were various suggestion about the evaluation method of interfacial strength that combined FEM analysis with microdroplet experiments, but there has been no study that simulated (2)~(4) at the same time while considering (1) in the past. Thus, this study suggests the accurate evaluation method of interfacial strength in microdroplet test by considering the above four points.

2. Experiments

2.1. Specimen

The epoxy resin droplets are attached to the single carbon fiber. This sample is put it an electric furnace, and the epoxy droplet is cured by baking 135℃ for 2 hours. A single fiber attached droplet is fixed to the jig (Fig. 1) by the tapes.

Fig. 1. Specimen

2.2. Experimental equipment

We putted the test machine (made by Shirasaki Corporation) on the stage of an optical microscope, and we measured the fiber load while observing the fiber and the resin (Fig. 2). The specimen is attached to the point of Cylinder and put a epoxy droplet between knife edges. Then a droplet is given load in the direction of the arrow of Fig. 2.

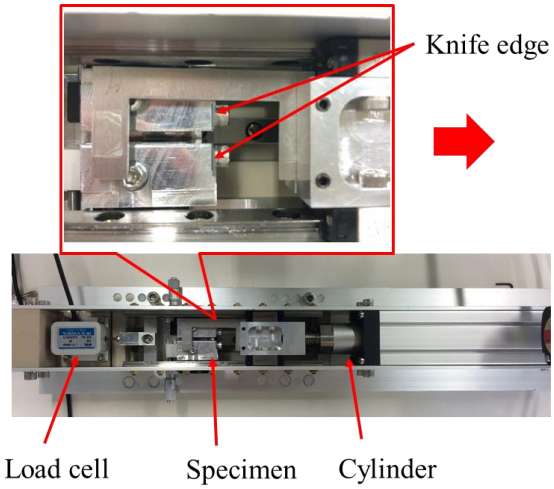

Fig. 2. Tensile testing machine

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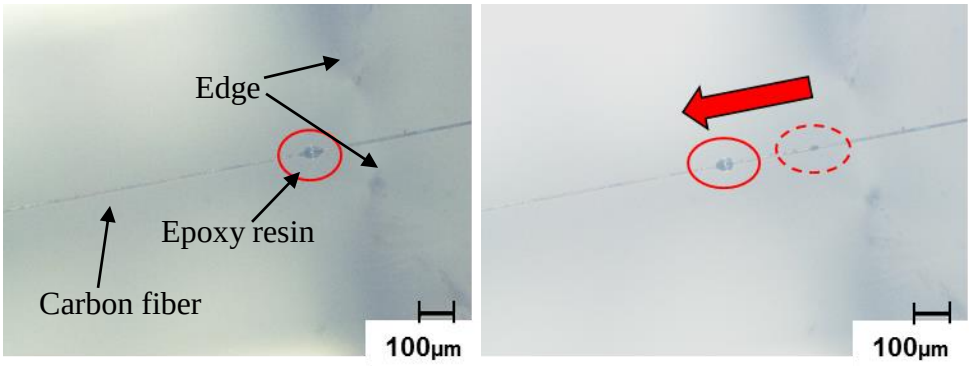
2.3. Experimental results

Fig. 3 is show before and after the specimen was loaded tensile load. The optical microscope image (b) is compared with (a), it is found that the positions of the epoxy droplet are different. In other words, the epoxy droplet was torn off by the edge and moved. Also, (b) shows that the scrap of the epoxy droplet remains on initial position (dotted line) and the epoxy droplet deformed. It was caused by loading to the epoxy droplet with edge.



(a) before experiment (b) after experiment

Fig. 3. Optical microscope image of microdroplet experiment

It is shown the time-load graph of interface between the fiber and the resin in this time, in Fig. 4. The peak load is 0.029N. The load after debonding was caused by the microdroplet that we could not see. This study determines interfacial strength by comparing experimental peak load with computational simulations of microdroplet tests.

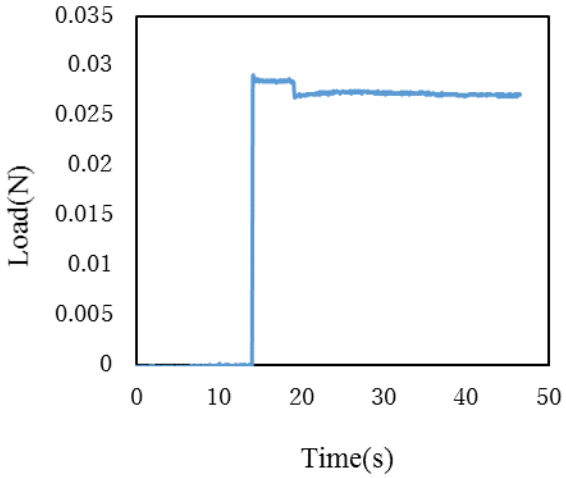


Fig. 4. The force of interface between the resin and the fiber

3. Analysis

3.1. Finite element model

Computational simulation is performed with the ABAQUS software package (Dassault Systèmes) using an axisymmetric finite element (FE) model patterned after the geometry shown in Fig. 1. This model has 3200 nodes, 1500 element and their elements are consisted of six node. The material properties are shown in Table. 1. Considering the heat-treatment of the specimen, this temperature gradient is 115℃. The contact analysis is in between the surface of resin and the surface of the edge.

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Cohesive elements (same as [17]) are introduced on the interface of the FE model and it is considered that compressive stress enhances interfacial shear strength. Also, dilatational damage and shear damage, based on continuum damage mechanics, are introduced into the resin.

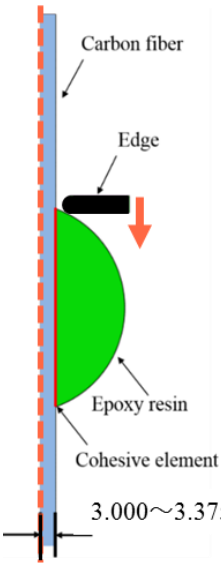

Fig. 5. Axisymmetric model

Table 1. Mechanical properties [15]

Fiber axial modulus	294 GPa
Fiber transverse modulus	14 GPa
Fiber axial Poisson's ratio	0.2
Fiber transverse Poisson's ratio	0.35
Fiber axial shear modulus	18GPa
Fiber transverse shear modulus	5GPa
Fiber axial thermal expansion coefficient	0
Fiber transverse thermal expansion coefficient	1×10^{-5}
Matrix modulus	4GPa
Matrix Poisson's ratio	0.34
Martix thermal expansion coefficient	5×10^{-5}

3.2. Thermal residual stress analysis

The relaxation modulus and time-temperature super position principal are considerd into the resin. The relaxation modulus are difined in eq.(1) [6]. In Equation (1), τ_c is relaxation time and n is power law component. Fig. 6 is the relaxation modulus caluculated by eq.(1).

$$E(t)=\frac{E_0}{\left(1+\left(\frac{t}{\tau_c}\right)^n\right)} \quad (1)$$

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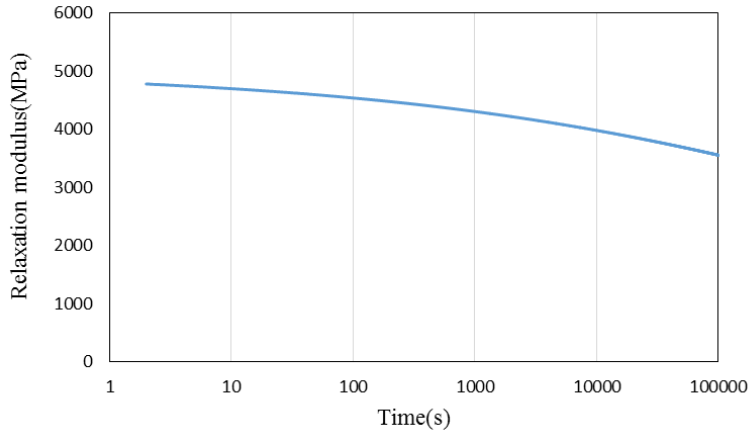

Fig. 6. Relaxation modulus [6]

The time accelerates when the temperature is high are considerd by time-temperature super position principal. Shift facter α is difined in eq.(2) with the activation energy ΔH by Arrhenius type and reference temperature T_R. The activation energy ΔH = 150 kJ/mol [6]. The reduced time is defined by eq.(3) and eq.(4) indicates stresses depended on temperature. In Equation(4), $\xi(t)$ is reduced time, θ is temperature ,$\tau(t)$ is shear stress, g_R is relaxation modulus and $\dot{\gamma}$ is strain rate.

$$\log \alpha = \frac{\Delta H}{2.303R}\left(\frac{1}{T} - \frac{1}{T_R}\right) \tag{2}$$

$$\xi(t) = \int_0^t \frac{ds}{\alpha(\theta(s))} \tag{3}$$

$$\tau(t) = \int_0^t g_R(\xi(t) - \xi(s))\dot{\gamma}(s)ds \tag{4}$$

3.3. Analysis result and discussion

Fig. 7 shows numerical results for the S12 stress distribution. When just after numerical simulation of thermal residual stress, the compressive stress are caused by thermal contraction on resin tips (Fig. 7a). The edge touches to the resin (Fig. 7b), and interface separation progressed when the cohesive elements failed (Fig. 7c). Fig. 7(d) is a magnified view of 7(c) and shows damage of the resin. Thus, we are able to express that the resin is damaged by the edge. Based the result, we change the cohesive element strength for adjusting the analysis result and the experimental result. As a result, the interfacial strength provided by numerical simulation is 60 MPa. As a reference, IFSS (Interfacial Shear Strength) that is calculated by eq.(5) is approximately 30 MPa. In Equation (5), τ is shear stress, F is the peak load, d is the diameter of the fiber, L is the embedded length. It is found that interfacial strength provided by analysis (cohesive element strength) is higher than IFSS, as it is supposed that the stress distribution of interfacial is constant in an interfacial domain in IFSS. Because the actual stress distribution is not constant in an interfacial domain and high stresses exists there, it might be higher than IFSS. These can be considered with cohesive element strength.

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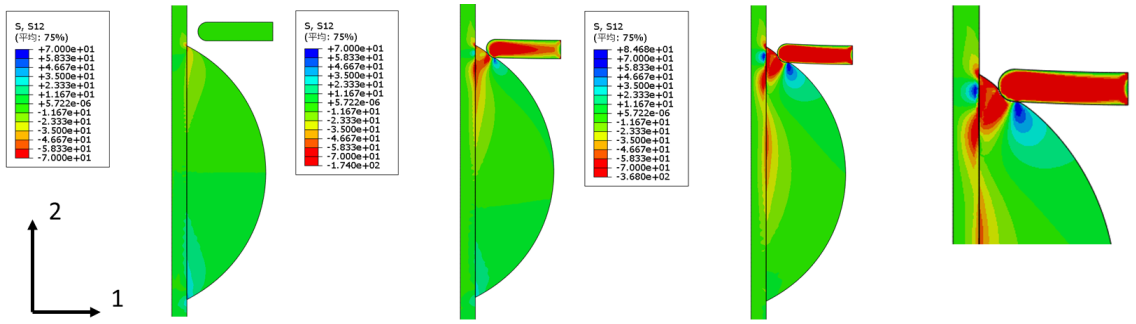

(a) (b) (c) (d)

Fig. 7. Numerical results for S12 stress distribution

(a) Just after numerical simulation of thermal residual stress, (b) The edge touch to resin, (c) After progressive debonding and (d) Magnified view of (c).

$$\tau = \frac{F}{\pi dL} \tag{5}$$

4. Conclusions

We measured the fiber-resin interfacial strength using the experiment of the microdroplet tests and the finite element analysis. In numerical simulation, we consider the accurate thermal residual stresses considered thermal viscoelastic, the damage of the resin when the edge touches the resin droplet, the interfacial debonding used cohesive elements and the transition of interfacial strength by the interfacial compressive stress. The interfacial failure and the damage analysis of the resin were carried out at the same time. The interfacial strength provided by numerical simulation is higher than IFSS.

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