

**Interfacial Adhesion Evaluation of Uniaxial Fiber-Reinforced-Polymer Composites by
Vibration Damping of Cantilever Beam**

by

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(Abstract)

The performance of fiber-reinforced composites is often controlled by the properties of the fiber-matrix interface. Good interfacial bonding (or adhesion), to ensure load transfer from matrix to reinforcement, is a primary requirement for effective use of reinforcement properties. Thus, a fundamental understanding of interfacial properties and a quantitative characterization of interfacial adhesion strength can help in evaluating the mechanical behavior and capabilities of composite materials. A large number of analytical techniques have been developed for understanding interfacial adhesion of glass fiber reinforced polymers. Common adhesion tests include contact angle measurements, tension or compression of specially shaped blocks of polymer containing a single fiber, the single fiber pull-out test, single-fiber fragmentation test, short beam shear and transverse tensile tests, and the vibration damping test. Among these techniques, the vibration damping technique has the advantage of being nondestructive as well as highly sensitive for evaluating the interfacial region, and it can allow the materials industry to rapidly determine the mechanical properties of composites.

In this work, we contributed a simple optical system for measuring the damping factor of uniaxial fiber-reinforced-polymer composites in the shape of cantilever beams. A single glass fiber- and three single metallic wire-reinforced epoxy resin composites were tested with the optical system. The fiber- (wire-) matrix interfacial adhesion strength measurements were made by microbond test. A reasonable agreement was found between the measured interfacial adhesion strength and micromechanics calculations using results from vibration damping experiments. The study was also extended to multi-fiber composites. The interfacial damping factors in glass-fiber

reinforced epoxy-resin composites were correlated with transverse tensile strength, which is a qualitative measurement of adhesion at the fiber-matrix interface. Four different composite systems were tested. For each system, glass fibers with three different surface treatments were used at three different volume fractions. The experimental results also showed an inverse relationship between damping contributed by the interface and composite transverse tensile strength for all of the multi-fiber composites.

To Mom and Dad

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CHAPTER 1

INTRODUCTION

Fiber-reinforced composite materials are being more widely used in many applications. The glass fiber composites have been used a great deal in chemical plants under hostile environments because of their good resistance to corrosion. They are also being used increasingly in automobiles, trucks, and other vehicles because of substantial weight reductions, and simplified manufacturing. With the increasing applications of these materials, more and more knowledge is needed to get a better understanding of the bonding of the materials, which can lead to different mechanical properties of the materials. It is also necessary to find a simple method to measure those behaviors.

Uniaxial fiber-reinforced polymers (FRP) are the simplest examples of composite materials. The properties obtained from these materials can be analyzed and modeled easily without considering other geometric parameters such as fiber length and fiber orientation. With a good knowledge of the properties for this type of material, an extension can be made to understand the properties of other composite materials.

It is well known that fiber-matrix interfacial adhesion plays an important role to improve mechanical properties of a composite. The tensile strength of the composite depends on the ability of the composite to effectively transfer tensile load from the matrix to the fiber via shear at the interface. Thus, a procedure to quantify the interfacial adhesion would contribute to an evaluation of the mechanical behavior of composite materials.

A large number of experimental techniques have been developed [1-14] for measuring interfacial adhesion in fiber-reinforced polymer composites. Among these techniques, vibration damping is promising because it provides a sensitive and nondestructive evaluation of the interfacial region, and can allow the materials industry to rapidly determine the interfacial adhesion strength of composites. Therefore, this non-destructive method would represent a valuable contribution in the evaluating mechanical performance of composite materials.

It is widely recognized that the interfacial bonding of a composite can be related to a change in energy absorbing ability, i.e., damping of the material. While theoretical and

experimental analyses are reported which support the validity of the concept [14, 15], there is presently no direct quantitative relation between interfacial damping and interfacial adhesion strength in the published work.

Therefore, the main objectives of this research are to: (1) develop a simple, noncontact optical technique based on vibration damping for measuring the interfacial adhesion of composites; (2) experimentally investigate the correlation between dynamic (vibration damping) adhesion measurements and static interfacial properties (interfacial adhesion strength) through an application to a specific series of fiber-reinforced polymer composites; (3) attempt to develop a model based on cantilever beam vibration to evaluate the adhesion between fiber and matrix from vibration damping data. The fiber volume fraction effect on the interfacial adhesion will also be studied.

This research is significant in that it not only provides a fundamental understanding of interfacial adhesion but also introduces a novel experimental technique. The interfacial adhesion between the fiber and matrix of a composite can be easily evaluated by a vibration damping technique without conducting tedious destructive experiments. With the relationship between the dynamic and static adhesion measurements, the interfacial adhesion strength, τ , can be easily obtained from the interfacial damping factor $\tan \delta_{in}$.

The thesis is organized as follows:

Chapter 2 presents a literature review with four topics to get a better understanding of the background of the present research. It contains a review of techniques used to measure fiber-matrix adhesion of composites, vibration damping concepts and mechanisms, damping in composite materials and earlier research on interfacial damping of fiber-reinforced polymer composites.

Chapter 3 describes sample preparation and the experimental system developed to measure the interfacial adhesion of composites.

Experimental results are presented in Chapter 4, which include both vibration damping and direct interfacial adhesion (fiber pull-out and transverse tensile tests) measurements.

Chapter 5 is the discussion. A micromechanics model for evaluating the adhesion between fiber and matrix from damping factor of a cantilever beam is described, in which a quantitative inverse relationship between damping factor contributed by the interface and interfacial adhesion

strength is established. The vibration damping measurements of single fiber-reinforced composites are compared with interfacial adhesion strength of the materials by the developed model. For real composites, interfacial damping data are compared with transverse tensile strength which is also a qualitative measurement of adhesion at the fiber-matrix interface.

Finally, in Chapter 6, the results of the present research are summarized.

CHAPTER 2

LITERATURE REVIEW

Theoretical and experimental work on interfacial adhesion measurements of fiber-reinforced polymer composites has been extensively pursued in recent years. This chapter provides a brief review of the commonly used techniques for measuring fiber-matrix adhesion. Due to the application of the vibration damping method in the present research, the general background of vibration damping is introduced and the mechanisms are discussed. The concept of interfacial damping which can be used to evaluate interfacial adhesion is also introduced.

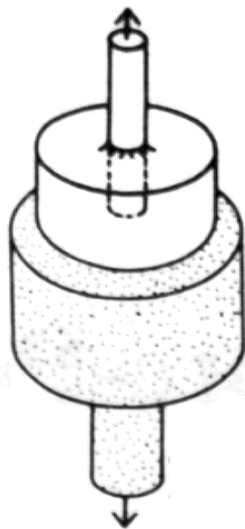
2.1. Measurement Techniques for Fiber-Matrix Interfacial Adhesion Strength

2.1.1. Destructive Methods

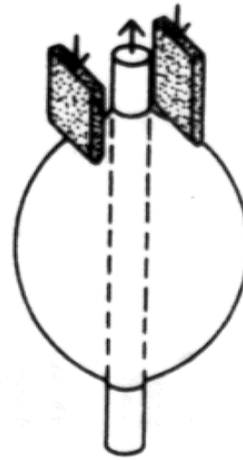
It is generally accepted that the adhesion between the fiber and the matrix has an effect on the mechanical properties of fiber-reinforced polymer (FRP) composites [16, 17]. In particular, the tensile strength of the composite is affected by efficiency of load transfer from the matrix to fiber via shear at the interface. Therefore, a number of mechanical tests have been developed to measure the capacity of the interface to transfer stress from the matrix to the fiber in a composite.

The interfacial properties were first measured with an indirect compression method suggested by Broutman [1] in 1966. With the improvement of techniques in the following years, many testing methods were applied. Currently, four techniques suitable for evaluating actual reinforcing fiber-matrix adhesion strength have become widely used. These mechanical tests were summarized by Piggott [2] and are illustrated in Figure 2.1.

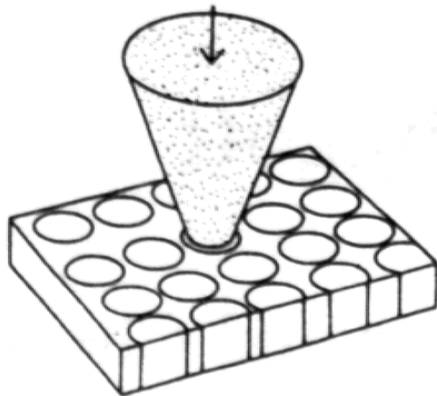
The pull-out [3] and microtension [4] (or called microbond pull-out) methods are basically equivalent. In the pull-out method, the fiber is embedded in a solid matrix material and pulled with an increasing force. A plot of force at the instant of debonding, F_d , versus embedded length, l , has been used to estimate the interfacial adhesion strength, τ , for fibers with a diameter $2r$:



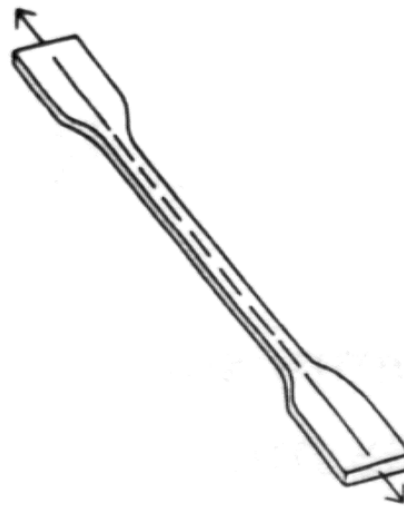
Pull Out



Microtension



Microcompression



Fragmentation

Figure 2.1 Four methods currently used for measuring interfacial properties. (adapted from reference [2])

$$\tau = \frac{F_d}{2\pi r l} \quad (2.1)$$

This test was originally applied to glass fiber-reinforced concrete and carbon filament-reinforced aluminum, and has been adapted by Favre and co-workers for use with FRP composites [5]. As demonstrated with many systems, it has been shown that the pull-out test is relatively easy to carry out with rod or thick fibers. However, when fiber-matrix interfacial adhesion is strong and fiber diameter is small ($< 10 \mu\text{m}$, for example), the embedded fiber length must be very small. Otherwise, it will result in the fiber breakage instead of pulling the fiber out of the matrix.

The microtension or microbond test was developed by Miller et al. [4] to prepare pull-out samples with very small embedded fiber length. In this method, the fiber is embedded in a small axisymmetrical drop of resin and pulled out. It is similar to the pull-out test, and F_d is plotted versus the bonded area or bond length to estimate the adhesion strength of the interface.

The microcompression or microindentation test was reported first by Mandell et al. [6]. This technique is becoming popular [7,8], which uses a small indenter to debond a fiber as shown in Figure 2.1. A composite specimen is sectioned perpendicular to the fiber axis, the end of a single fiber is compressively loaded with a very fine diamond tip, and the fiber is pushed down from the composite. With a finite-element analysis it is possible to estimate the shear stress at the instant of debonding.

The single fiber fragmentation test [9] has been used widely in recent years [10]. In this method, a single fiber is embedded in a polymer resin with a mold to form a dogbone shape tensile specimen. When the dogbone specimen is stretched in the fiber axial direction, the fiber breaks into small fragments, until finally the fiber lengths are all less than a critical length, l_c . Acoustic emission provides a reliable means for determining the number of breaks, and hence the average value of l_c [11]. The interfacial adhesion stress, τ , is estimated from l_c assuming that it is constant along the fiber length.

$$\tau \approx \frac{3\sigma_{fu} r}{4l_c} \quad (2.2)$$

where σ_{fu} is the fiber strength. Weibull statistics are usually applied to estimate σ_{fu} [12].

Each of the methods mentioned above has inherent problems that limit its applicability as a universal test method for characterization of interfacial adhesion strength [13]. All of these

techniques deal with individual fibers but not fibers in a real composite and may not reflect the overall interfacial property of the composites. Due to the complexity of the behavior of the interface, it is not clear at the present time that a correlation exists between the results obtained among the test methods described above. Therefore, when the interfacial adhesion strength is mentioned, it is usually reference to a specific test method.

2.1.2. Non-Destructive Methods

Besides those destructive methods mentioned above, some nondestructive evaluation (NDE) techniques [18] such as radiography, acoustic emission, thermal NDE methods, optical methods, vibration damping techniques, corona discharge and chemical spectroscopy, have also been applied to characterize the fiber-reinforced composites. Among these techniques, the vibration damping method, which is based on energy dissipation theory, has been increasingly used for measuring interfacial adhesion. The principle of the method is based on the theory of energy dissipation. According to the theory, quality of interfacial adhesion in composites can be evaluated by measuring the part of energy dissipation contributed by the interfaces, assuming that the interface part can be obtained by separating those of matrix and fiber from the total composites. The energy dissipation of a material can be evaluated by the damping of the material.

Nowick and Berry [19] summarized the techniques currently used for measuring vibration damping of materials and structures. The techniques for the measurement of damping often deal with natural frequency or resonant frequency of a system. In general, all apparatus for the investigation of vibration can be categorized as free vibration (or free decay) and forced vibration.

Free vibration is executed by a system in the absence of any external input except the initial condition inputs of displacement and velocity. For example, it is possible to have a wire sample gripped at the top, and have a large weight hanging freely at the bottom; this system can be set either into longitudinal or torsional oscillation. The latter represents the well-known “torsion pendulum”, developed by Kê [20] in which the strain at any point can be expressed in terms of the angular twist of the inertia member.

For a forced vibration, a periodic exciting force is applied to the mass. When the resonant frequency is achieved, the loss angle δ is obtainable directly from the width of the resonance peak

at half-maximum in a plot of (amplitude)² versus frequency. Typical forced vibration techniques include the free-free beam technique [21] and the piezoelectric ultrasonic composite oscillator technique (PUCOT) [22-24]. These techniques have been applied to dynamic mechanical analysis (DMA) which is a widely used technique in polymer studies, and has attracted even more attention for interface characterization. However, the instrument is relatively expensive and can not be operated at a high frequency which can reflect more information from the tested materials.

Because vibration damping is increasingly being applied to characterize interfacial adhesion, it is necessary to understand the origin of damping. In the following section, a brief description of currently known damping mechanisms will be introduced.

2.2. Vibration and Damping

2.2.1. General Relation and Definition

Damping in principle is the mechanism by which the ordered mechanical energy of a material or a system is dissipated as disordered thermal energy to its surroundings as an irreversible process. It is observed that all free vibrations cannot keep going indefinitely and will die out ultimately. In other words, there is some resistance to the motion of the body.

Gemant [25] gives an excellent summary of vibration damping. According to his analysis, a simple model for describing forced vibration of a body with internal resistance is given by the following general differential equation based on Newton's second law:

$$F - c\left(\frac{dx}{dt}\right) - kx = m\left(\frac{d^2x}{dt^2}\right) \quad (2.3)$$

Here t represents time and x is the time-varying displacement; F is the external force. The second term on the left-hand side of Equation (2.3) is the damping term which is ideally considered as viscous damping, and the constant of proportionality c is called the viscous damping coefficient; the third term on the left-hand side of the equation is the elastic term, k is the stiffness, depending on the elastic modulus and a shape factor; the term on the right-hand side of the equation is the inertia force, and m is the mass of the body. The natural frequencies can be found without consideration of the damping effect in the case of free vibration, and the amplitude vs. frequency characteristic in most cases of forced vibrations.

The damping term, introduced into Equation (2.3), can be explained as follows.

If a body undergoes free vibration, i.e. there is no F term in Equation (2.3), the differential equation can be solved to lead to the following expression for the amplitude x [25]:

$$x = x_0 \exp(-\zeta \omega_0 t) \cos(\omega_d t - \phi) \quad (2.4)$$

Here x_0 is the amplitude at time $t = 0$; ω_0 is the natural frequency; ϕ is the phase angle; ω_d is called the dynamic frequency and is defined as $\omega_d = (1 - \zeta^2)^{1/2} \omega_0$; ζ is the damping factor that is related to the logarithmic decrement d and will be explained later:

$$\zeta = d / \sqrt{(2\pi)^2 + d^2} \quad (2.5)$$

When damping is small,

$$\zeta \cong d / 2\pi \quad (2.6)$$

The decay of the vibration is exponential according to Equation (2.4), which was verified by observations. In reality, ζ depends on the initial amplitude of the deformation, but it is still possible to obtain an average value of ζ from the decay curves.

As just introduced above, another quantity, the logarithmic decrement d is often used in preference to ζ ; it is the logarithm of the ratio of amplitudes one period of vibration apart. If n is the number of cycles of the vibration, x_0 is the amplitude of the first vibration, and x_n is amplitude of the n^{th} vibration, then [25]

$$d = \frac{\ln(x_0 / x_n)}{n} \quad (2.7)$$

It is known that the energy of vibration is proportional to the square of the amplitude, the relative loss of vibrational energy ($\Delta W/W$) is then given by

$$\Psi = \frac{\Delta W}{W} = 2d \quad (2.8)$$

where ΔW is the energy dissipation in a cycle and W is the maximum stored elastic energy of the system. Ψ is called the specific damping capacity which has an easily visualizable meaning.

Another way to find ΔW and W is the concept of complex modulus which can be physically explained by considering the phase difference between sinusoidally varying stress and strain for uniaxial loading [26]. According to the basic hysteresis response in a structure which is

subjected to an alternating force, an alternating strain lags behind the corresponding alternating stress by a phase angle δ , see Figure 2.2.

For a periodic stress imposed on a specimen, the relation for stress σ and strain ϵ are given by

$$\sigma = \sigma_0 \exp(i\omega t) \quad (2.9a)$$

$$\epsilon = \epsilon_0 \exp[i(\omega t - \delta)] \quad (2.9b)$$

where σ_0 and ϵ_0 are the stress and strain amplitudes, $\omega = 2\pi f$ is the circular (or angular) frequency (f is the vibrational frequency). In a perfectly elastic material, $\delta = 0$ and $\sigma / \epsilon = E$, i.e., the elastic modulus. However, most materials are anelastic, so δ is not zero and the ratio σ / ϵ is complex. This complex modulus may be expressed as

$$E^*(\omega) = \sigma / \epsilon = |E(\omega)| \exp[i\delta(\omega)] \quad (2.10)$$

where $|E(\omega)|$ is the absolute dynamic modulus. The complex modulus may also be expressed as

$$E^*(\omega) = E'(\omega) + iE''(\omega) \quad (2.11)$$

where $E'(\omega)$ and $E''(\omega)$ are the real and imaginary parts of $E^*(\omega)$, respectively. From the appropriate vector diagram, it is easy to show that

$$|E|^2 = E'^2 + E''^2 \quad (2.12a)$$

$$\text{and} \quad \tan \delta = E'' / E' \quad (2.12b)$$

Alternatively, E' and E'' are also called the storage modulus and loss modulus, respectively, and they are related to the energy stored, W , and energy dissipated, ΔW , per cycle of vibration by

$$W = \int_{\omega t=0}^{\omega t=\pi/2} \sigma d\epsilon = \frac{1}{2} E' \epsilon_0^2 \quad (2.13a)$$

$$\text{and} \quad \Delta W = \oint \sigma d\epsilon = \pi E'' \epsilon_0^2 \quad (2.13b)$$

where the stress-strain curve is assumed to be linear. Hence, the specific damping capacity (Ψ), or SDC, is given by

$$\Psi = \Delta W / W = 2\pi(E'' / E') = 2\pi \tan \delta \quad (2.14)$$

$\tan \delta$ sometimes is also called the loss coefficient, η . By comparing Equations (2.8), (2.14) and (2.6), the following relationship of different damping characteristics can be easily derived [25, 27]:

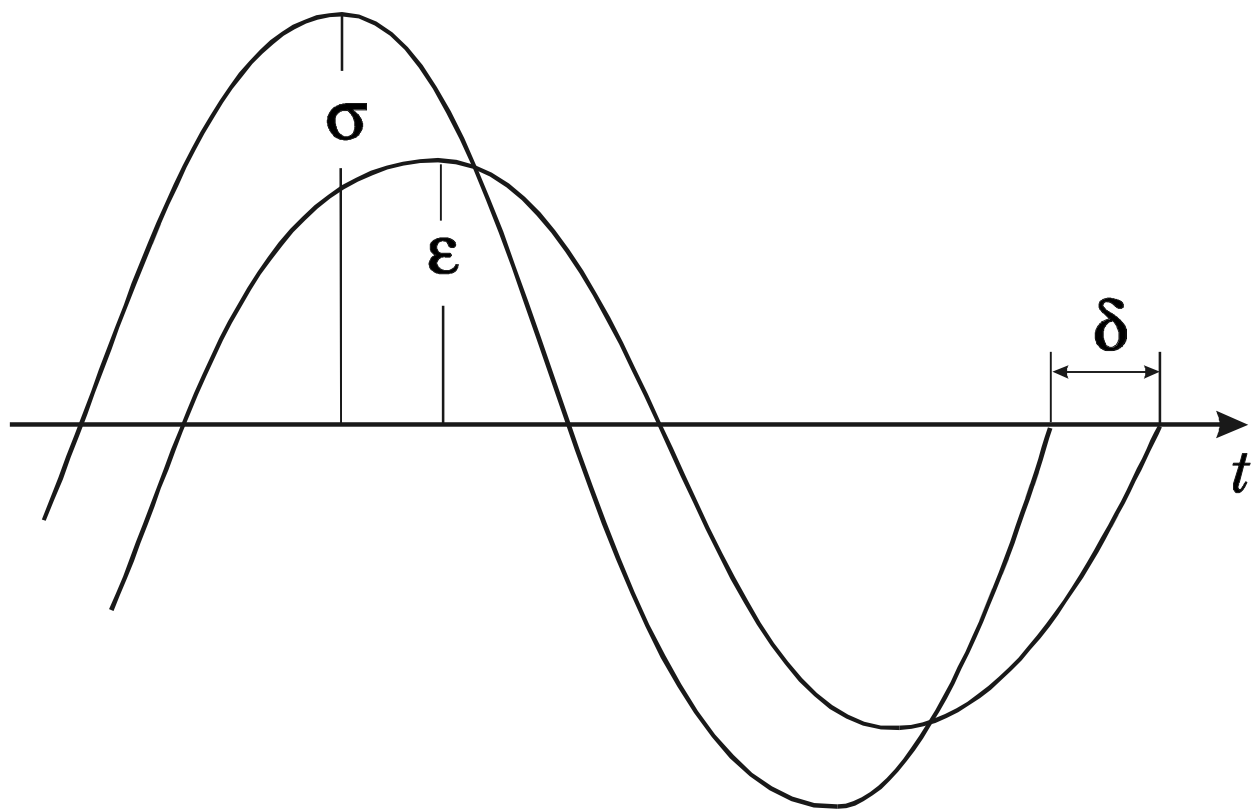


Figure 2.2 Phase-lag between stress and strain.

$$\tan \delta = \eta = \frac{d}{\pi} = 2\zeta = \frac{\Psi}{2\pi} = \frac{\Delta W}{2\pi W} \quad (2.15)$$

2.2.2. Beam Vibration and Damping

There are several ways to set up the vibration damping experiment in practice. A frequently used method is to gather the experimental data concerning dynamic material behavior by studying the free and forced transverse vibrations of cantilever beams made of the material. The information on beam behavior collected in the experiments was interpreted by comparing it with the behavior of a uniform, homogenous Bernoulli-Euler cantilever beam, vibrating in its various principal modes. The general response of the vibration is similar to Equation (2.4) [28, 29].

The widely accepted experiment was done by detecting the response of the cantilever beam excited by a vibration exciter. Resonant frequencies were determined by observing the peaking of response with varying excitation frequency at constant excitation amplitude. Free vibration decay measurements were usually made by exciting the beam at a resonant frequency, cutting power to the exciter, and analyzing the decay trace.

If the cantilever beam is excited in another way, such as deflected with a pin (i.e., a pre-displacement is given), then the pin is retracted to initiate vibration. The frequency and damping ratio can also be obtained by analyzing the decay trace. However, there are some problems with this type of set-up. The reasons are explained as follows.

When a cantilever beam is excited by a pre-displacement, the beam is vibrated with a frequency which is primarily composed of its first mode of vibration, and it can be understood by noting the vibrating shape (refer to Figure 2.3). However, the response is the linear combination of all of the modes because it is not vibrated with an exact resonant frequency, otherwise it is not a frequency controlled exciting.

2.2.3. Damping Mechanisms

As it was stated in section 2.2.1, damping in principle is the mechanism by which the ordered mechanical energy of the material or the system is dissipated as disordered thermal energy

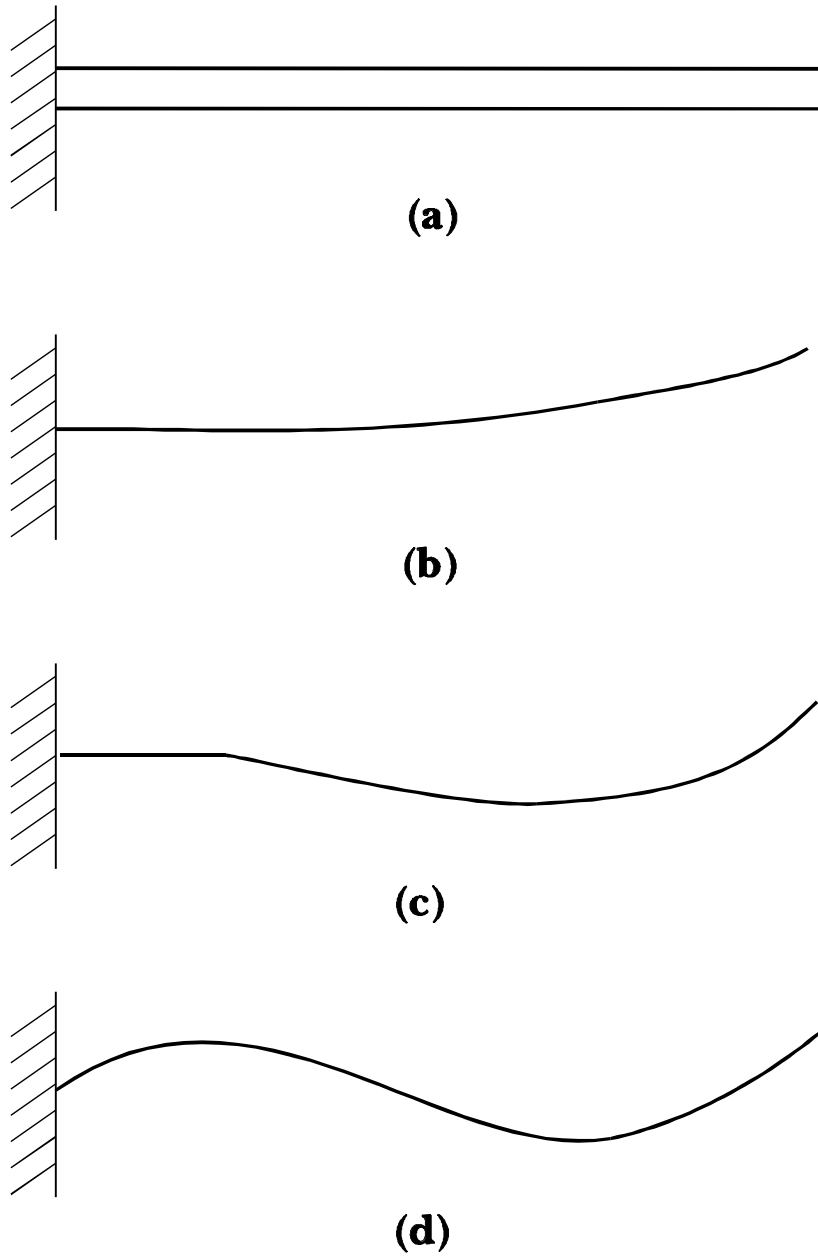


Figure 2.3 Mode shape of vibrated cantilever beam. (a) Cantilever beam; (b) Mode 1; (c) Mode 2; (d) Mode 3. (adapted from reference [29])

to the environment in an irreversible manner. The origin and mechanism of damping are complex and sometimes difficult to comprehend. There are several theories to explain the phenomenon, which can be divided into two categories, i.e., macro- and micro-mechanisms. In the following description, all possible damping mechanisms will be introduced first, and then the attention will be focused on those of polymers and their composites.

(1) Macro-mechanisms

The macro-mechanisms are those that involve the entire process of the vibration damping. It is known that material damping represents a departure from ideally elastic behavior, which implies that the strain induced by an applied stress contains contributions of either a plastic or viscoelastic nature. In fact, damping is closely related to internal friction of the material which is also a measure of energy dissipation in the material. It appears that there are two entirely different processes that lead to losses during mechanical vibrations. One is a thermal process, and another one is a strict consequence of the plastic flow of solids. Both appear to be general and of about equal significance.

Generally speaking, thermal losses will occur in inhomogeneous materials, such as in polycrystalline metals which consist of a large number of grains. These losses occur only if the material has volume changes; bending and longitudinal vibration usually cause the losses. In the case of pure shear (torsion), these losses will not exist. It has been shown [30, 31] that, d , due to thermal loss, should be independent of the strain amplitude ϵ_0 .

Plastic losses, on the other hand, will occur in any material, but presumably most readily in single crystals in which slip occurs easily, and also in materials of low creep resistance such as lead, and the whole group of organic plastics. These losses occur independent of the nature of strain. Either compression or shear can cause the losses. With increasing temperature these losses obviously will be favored. d increases with increasing the strain amplitude. This mechanism is frequency dependent; generally there should be a decrease of d with increasing frequency.

A. Macro-thermoelasticity

The thermoelastic effect is thermodynamically convertible, which is just like the

phenomenon of thermal expansion. When a material is rapidly deformed under an inhomogeneous stress, local temperature differences are produced depending upon the local stress levels and properties of the material. Consequently, temperature gradients formed within the material tend to produce heat flow unless the load is quickly released. Under high-frequency vibration, the time for significant heat flow is insufficient and the process remains adiabatic and reversible; thus, the observed damping is small. On the other hand, if the frequency is very low, the process is still reversible because it remains isothermal. Therefore there is still no significant dissipated damping energy. However, when a critical frequency is reached, the period of the cyclic stress is comparable with the time required for significant heat flow along the temperature gradients produced, an irreversible conversion of mechanical energy into heat occurs and damping will be observed.

Macro-thermoelastic damping occurs in many types of materials and vibrations. For example, damping of a German silver* beam was tested under flexural vibration, both the experimental points [32] and the theoretical curve [33] illustrate the general shape of the damping or relaxation peak. Similar thermoelastic effects can also be found in longitudinally vibrated bars [34].

B. Plastic flow mechanism

It is evident that in the case of vibrations, a plastic flow accompanied with elastic displacements must result in damping. This effect can easily be visualized by the so-called hysteresis loop.

Figure 2.4 shows the stress-strain diagram of a solid material under vibration. The straight line *AB* represents small amplitudes vibration at which plastic flow is negligible, and there is no hysteresis. For larger amplitudes the closed curve *CDEF*, which is run through in clockwise direction, represents the vibration cycle. Because of the permanent plastic deformation, strain is no longer a single value function of the applied stress. There are two different strains for any value of the stress. Therefore, for zero stress at *C* there is a residual strain *OC* from the previous

* The American Society for Testing Materials, under classification of Cased Copper-Base Alloys (B 119), classifies these alloys as having additions of over 10 percent of zinc, nickel in amounts sufficient to give white color, and lead under 0.5 percent.

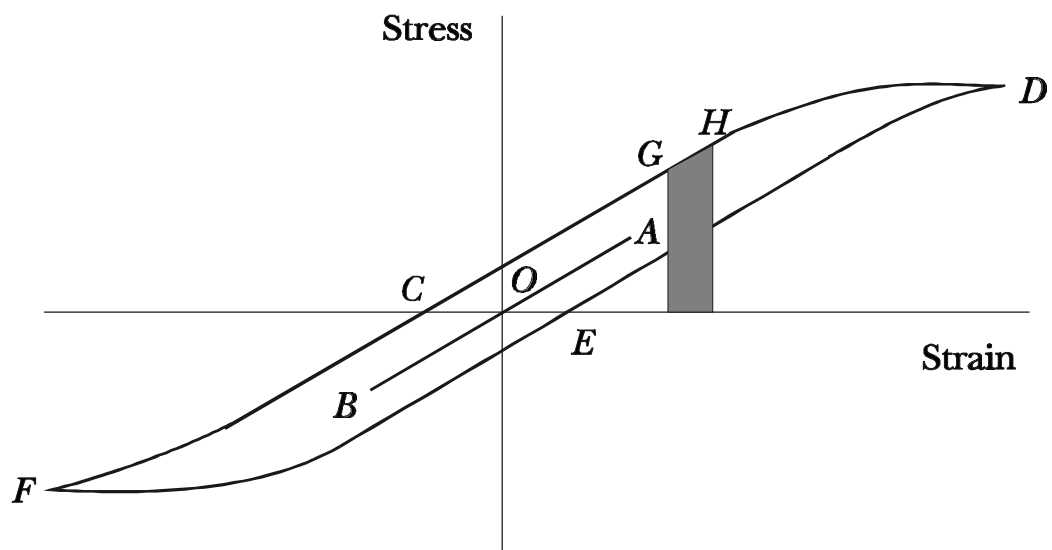


Figure 2.4 Hysteresis loop. (adapted from reference [25])

cycle, and after another half-cycle when the stress becomes zero again, the residual strain becomes OE . The shaded area along the element GH represents the element of work that the external stress has to do. The total work done during one complete cycle can be obtained by integration.

The area covered by the loop is the hysteresis loss for the complete cycle. If it is assumed that the shape of the loop does not depend upon the vibrating speed, then the hysteresis loss may be considered independent of the frequency. The loop can be identified with the specific loss, or the loss per cycle for alternating vibration, denoted by ΔW as mentioned above. From Equation (2.7) the logarithmic decrement d can be immediately obtained.

(2) Micro-mechanisms

When further consideration is taken of the damping mechanisms, it is necessary to consider the internal structures of materials which can have great effect on the energy dissipation because defects of materials are obviously influenced by the applied stress. After continuous study and measurement by metallurgists and physicists on the details of single crystal and polycrystalline materials [35, 36] some common defects that provide mechanisms for damping were proposed, which include concentration of point defects (Snoek [37] and Zener [38] relaxations), dislocation motion, grain boundaries, phase transformations, and differential temperatures between the specimen and its surroundings or between one part of the specimen and another part (thermalelastic or Zener damping).

A. Point Defect Damping

Micrographically, crystalline materials generally have many types of defects (vacancies or extra atoms) or impurities [31] which produce a local non-symmetry in the crystal. As a consequence, it will reduce the symmetry of the crystal; crystallographically equivalent positions or sites in the crystal will be occupied by the point defects. This type of imperfection is termed defect symmetry and leads to anelastic behavior. Under cyclic stress such "defective microstructures" will dissipate energy and produce hysteretic loop or damping effects. These operative micromechanisms have been identified and equations defining their characteristics have been developed by Nowick [31].

B. Grain Boundary Damping

Grain boundaries of a polycrystalline material are in a relatively disordered state and display viscous-like properties. Therefore, the unit energy dissipated in grain boundaries under cyclic shear stress or strain is larger, in general, than that dissipated within the grain [39]. The damping associated with grain boundary shear thus depends on the shear properties of the grain boundary which is a function of temperature, and also of strain rate (or frequency).

If only the shear effects are considered, according to the definition, the energy dissipated by grain boundary migration is proportional to the product of the shear stress and anelastic shear strain. When the vibration frequencies are high or the temperature is low, the shear stress across a grain boundary can relax only slightly during a vibration cycle and material still remains elastic. Thus, although the shear stress may be large, the anelastic shear strain is small, and the hysteresis loop is narrow, small damping will be observed. By contrast, when the vibration frequencies are low or the temperature is high, large stress relaxation can occur under cyclic strain and the shear stress is correspondingly reduced. In this case, the area within the hysteresis loop is still small. However, at some intermediate condition, the rate of stress relaxation is optimum for producing the maximum area within the hysteresis loop, the maximum damping will be observed.

C. Dislocation Damping

Dislocation damping plays an important role in crystalline materials. When a crystalline material is deformed under an alternating stress, the dislocation movement may lag behind the applied stress. As a sequence a hysteretic loop will be formed, which is contributed to the energy dissipation. If it is assumed that the dislocations are perfectly mobile without any restriction on their motion, the estimated magnitude of damping effect for typical dislocation densities would be much larger than that observed experimentally. Therefore, impediments to the motion of dislocations or other types of interactions must be considered. Such impediments can be pinning points, network points, jogs, and other dislocations.

As mentioned above, in order to dissipate energy the dislocation motion must lag behind the applied stress. One generally accepted dislocation model for damping was proposed by Koeher [40] and further developed by Granato and Luecke [41] (termed G-L model hereafter).

In this model dislocation motions are viewed as being analogous to a vibrating string in a viscous medium. By considering the effective mass per unit length and the effective tension in the dislocation, a differential equation for the model can be written as [42]

$$A \frac{\partial^2 y}{\partial t^2} + B \frac{\partial y}{\partial t} - C \frac{\partial^2 y}{\partial x^2} = b\sigma \quad (2.16)$$

where A is the effective mass per unit length, B the viscous damping constant, C the tension force, b the Burgers vector and σ the applied stress.

At low frequencies, the velocity of the dislocation is small, which means that the viscous force and damping are small. In these cases the displacement, y , is limited by the tension forces in the dislocation and it appears parabolic in shape. As the frequency increases, the viscous forces become dominant and the displacement of dislocation cannot achieve its full parabolic values. Under these circumstances it behaves like a rigid rod except for curvature at its ends near the pinning points. At very high frequency the drag forces may be large but the displacement is very small, therefore the damping is still small. At some intermediate or optimum frequency the integral of the drag forces and displacement is maximum and thus the damping is maximum.

D. Thermoelastic Damping

Thermal currents not only lead to damping macroscopically, but also they can exist on a microscopic scale. This phenomenon was observed in polycrystalline materials.

It is well known that in a polycrystalline material, individual crystals are generally anisotropic with different thermal and mechanical properties in the direction of principal stresses. Local temperature gradients are therefore produced even though the material is under macroscopically uniform axial stress. If a cyclic stress is applied to the material, this microanisotropy can cause cyclic micro-thermal currents across grain boundaries. This process also causes some energy dissipation [35]. At certain combinations of temperature and frequency the energy dissipation effects are maximized. In other words, the damping peak or the relaxation peak can be observed.

(3) Damping Mechanisms in Polymers

In the foregoing introduction, the damping mechanisms are mainly dealt with metallic materials. What are, however, the damping mechanisms of nonmetallic materials such as wood, stone, glass, and (especially) polymers? So far, little is known about the important micromechanisms in most nonmetallic materials.

In fact, much work has been done on polymers although little is known about damping mechanisms of most other nonmetallic materials because of their complex structure.

It is known that with larger molecules and those with long-range molecular order, polymers behave with both elastic and viscous characteristics. This means their properties are intermediate between that of a crystalline solid and a simple liquid. By changing the monomeric units, the molecular weight, degree of branching and crosslinking, the main chain stereo configuration of monomers, and other features of the molecular chemistry, the desired mechanical properties can be obtained. High damping is an important behavior of polymers. It has been known that damping in most polymers is dependent on frequency and temperature. A critical temperature, T_g , and frequency, ω_c , at which a polymer has the maximum damping is always observed [26].

Kaelble [43] described the micromechanisms of damping in polymers based on microstructure and phenomenology, and classified them into three basic categories: secondary transition; glass transition; melting and flow.

It is well known that at temperatures near absolute zero, the main source of motion in polymers is provided by the bending and stretching of primary valence bonds. When temperatures increase, but still below the glass transition temperature T_g at which the polymer undergoes the transformation from a rubber to a glass, side groups can move and will cause dynamic dispersions. The magnitude of the peaks in $\tan \delta$ in this case are usually smaller than those obtained at T_g , and such phenomena are called as “secondary transitions” and are associated with the character of the monomeric structure of the polymer.

There is particularly strong damping at the glass transition temperature, T_g . This critical temperature is governed by the main chain in the amorphous phase of the polymer. The length of the chain segment is small compared that of macromolecule but is larger than the monomer group. Segment motion involves the coiling and uncoiling of sections of the macromolecular chain. It has been found that significant damping occurs at T_g when the applied test frequency equals the

natural frequency for main chain rotation. If frequencies are higher, the time for chain uncoiling is insufficient, and the polymer will appear to be relative stiff. When frequencies are lower, the time for chain movement is larger and the material will be soft and rubbery.

Between the glass transition temperature T_g and the crystalline melting temperature T_m , the damping response of polymers is insensitive to temperature. The high-temperature limits of this elastomeric region of response is determined by the melting and flow properties of the macromolecule. If the macromolecule is incorporated into a three-dimensional crosslinked network, the high-temperature limits of elastomeric response are restricted only by the thermal and environmental stability of the chemical valence bonds.

2.3. Composite Materials and Their Damping

In the foregoing section, damping mechanisms of common materials have been discussed. These can be referred to as intrinsic mechanisms. However, if two or more materials are combined together to form a composite, the properties of the composite are governed not only by the properties of individual components but also by the interface between them. When the composite undergoes vibration damping, additional mechanisms, known as extrinsic mechanisms, will be introduced.

2.3.1. Composite Materials

Composite materials fall into two categories: fiber reinforced and particle (or whisker) reinforced composite materials. Both are widely used in advanced structures. Among the various kinds of composites, glass fiber-reinforced polymer (GFRP) composites have become more and more important in engineering applications because of their low cost, light weight and good corrosion resistance. Successful reinforcement of these composite materials is only achieved by obtaining sufficient load transfer between fiber and matrix. However, an extremely strong interfacial bond is not always desirable. In some cases, a weak interface is desirable to promote fiber pull-out, hence providing a tougher composite system. It would also be desirable to design the interfacial properties to suit a particular application.

In the first section of this chapter, the existing techniques for measuring interfacial adhesion have been reviewed. Among those techniques, it has been shown that vibration damping is promising because it provides a sensitive and nondestructive evaluation of the interfacial region. In the next section the principle of the technique will be discussed.

2.3.2 Damping in Composite Materials

The damping of fiber reinforced composite materials has been studied extensively [44, 45]. All of the published results for continuous fiber reinforced composites show that when strain levels are low the damping characteristics do not depend on strain amplitude but are dependent on fiber orientation, temperature, moisture absorption, frequency, and matrix properties. Fiber properties have only minimal effects. However, for discontinuous fiber reinforced composites it has been shown that the damping characteristics in the fiber direction are much greater than that obtained continuous fiber reinforced composites.

It is commonly accepted that the main sources of damping in a composite material come from microplastic or viscoelastic phenomena associated with the matrix and slippage at the interface between the matrix and the reinforcement. Therefore, besides the contribution from any cracks, debonds and the orientation of the reinforcement, composite damping will be affected by the following factors:

1. the properties and the volume fraction of matrix and reinforcement in the composite;
2. the size of the reinforcement;
3. the surface treatment of the reinforcement.

It is clear that the explanation of damping in composite materials is a very complex matter, to which much research has been dedicated.

2.3.3. Interface Effects in Composites

As mentioned in the previous section, one unique characteristic of a composite is its large internal interfacial area. Thus, in analyzing the damping of a composite it is important to consider not only the individual component materials but also the energy dissipation caused by the interface. Therefore, the damping analysis of a composite generally requires a recognition of the

individual components in the composite and a determination of their contributions to the total composite [46].

Thus, extrinsic damping mechanisms in a composite are mainly related to the reinforcement-matrix interfaces. For a poorly bonded interface, the damping can be caused by a sliding friction mechanism (Coulomb friction). On the other hand, if an interface is well-bonded, it could also lead to increased damping via an increased dislocation density near the fiber-matrix interface although this mechanism usually is not valid for polymer matrix composites because non-crystallized polymers are applied in most cases.

2.4. Earlier Research on Interfacial Damping of Fiber-Reinforced Polymers (FRP)

In this section a review of papers on interfacial damping in FRP will be presented in order to define the objectives of the present research.

One way to define interfacial bond in a composite mathematically is that it has zero thickness and is the result of the interaction between fiber surface and matrix. It can be divided into three levels: weak, ideal and strong. Generally speaking, ideal interfaces simply play the role of effectively transferring load and do not contribute to damping. In such a case, the composite damping term ($\tan \delta_s$) can be estimated from the rule of mixtures (ROM). For a reinforcement with low damping, $\tan \delta_f = 0$ and Nielsen [47]. suggested that:

$$\tan \delta_s = (1 - V_f) \tan \delta_m \quad (2.17)$$

where $\tan \delta_s$ is the effective loss tangent for a composite without consideration of interfacial adhesion, V represents volume fraction. Subscripts m and f refer to the fiber and matrix, respectively.

In practice, the interface region often participates in the damping of composites. Depending on composite type and the level of interfacial bonding, different approaches have been proposed to estimate the contribution of the interfaces to the damping of the composites. According to the theory of energy dissipation, the quality of the interfacial adhesion in composites can be evaluated by measuring the part of energy dissipation contributed by the interfaces; the interface part can be obtained by separating the matrix and fiber from the total composites. Zorowski and Murayama [14] were the first to develop a method for measuring the quality of the

interfacial adhesion in reinforced rubber through energy dissipation measurements based upon the following relationship:

$$\tan \delta_{in} = \tan \delta_c - \tan \delta_s \quad (2.18a)$$

$$\tan \delta_s = \frac{\tan \delta_f E_f V_f + \tan \delta_m E_m V_m}{E_c} \quad (2.18b)$$

where E is the Young's modulus, subscript c refers to composite. $\tan \delta_{in}$ is the internal energy dissipation from interface, and $\tan \delta_c$ is the measured internal energy dissipation of the composite. By measuring the total system energy dissipation in terms of $\tan \delta$ and knowing $\tan \delta$ and the dynamic moduli of the components as well as the volume fraction of fibers, the dissipation due to poor interfacial adhesion can be determined. From experimental work done by Murayama et al. [15], the energy loss at interfaces was also found to directly relate to the strip adhesion peeling load for nylon and PET fiber reinforced vulcanized rubber composites. The higher the damping contributed by the interface, the weaker the interfacial adhesion. This result is shown in Figure 2.5.

Much work has been carried out to correlate damping characteristics with interfacial structure and properties.

Gibson and Plunkett [48] studied crossly laminated glass-epoxy composite beams by using steady-state forced vibration tests. The results showed that the damping increases slightly with increasing maximum strain during the first run of the vibration. When the strain level is reduced, the damping remains near the same indicating permanent structure change. They attributed the phenomenon to Coulumb friction at the newly-created crack interfaces.

By introducing cracks in composite samples, Adams et al. [49] found that damage on specimens fabricated from fiber reinforced polymers could be detected by damping increasing whether this damage is localized as a crack, or distributed through the whole specimens as many microcracks. They also studied dynamic torsion of specimens with high volume fraction of fibers at different amplitudes [50], and demonstrated a significant increase in damping of the composites with increasing strains. The results were explained as the propagation of the delaminated cracks.

In order to improve the interfacial adhesion, surface modification of reinforcing fibers has been extensively applied in industry. Cinquin et al. [51] introduced a coupling agent at the

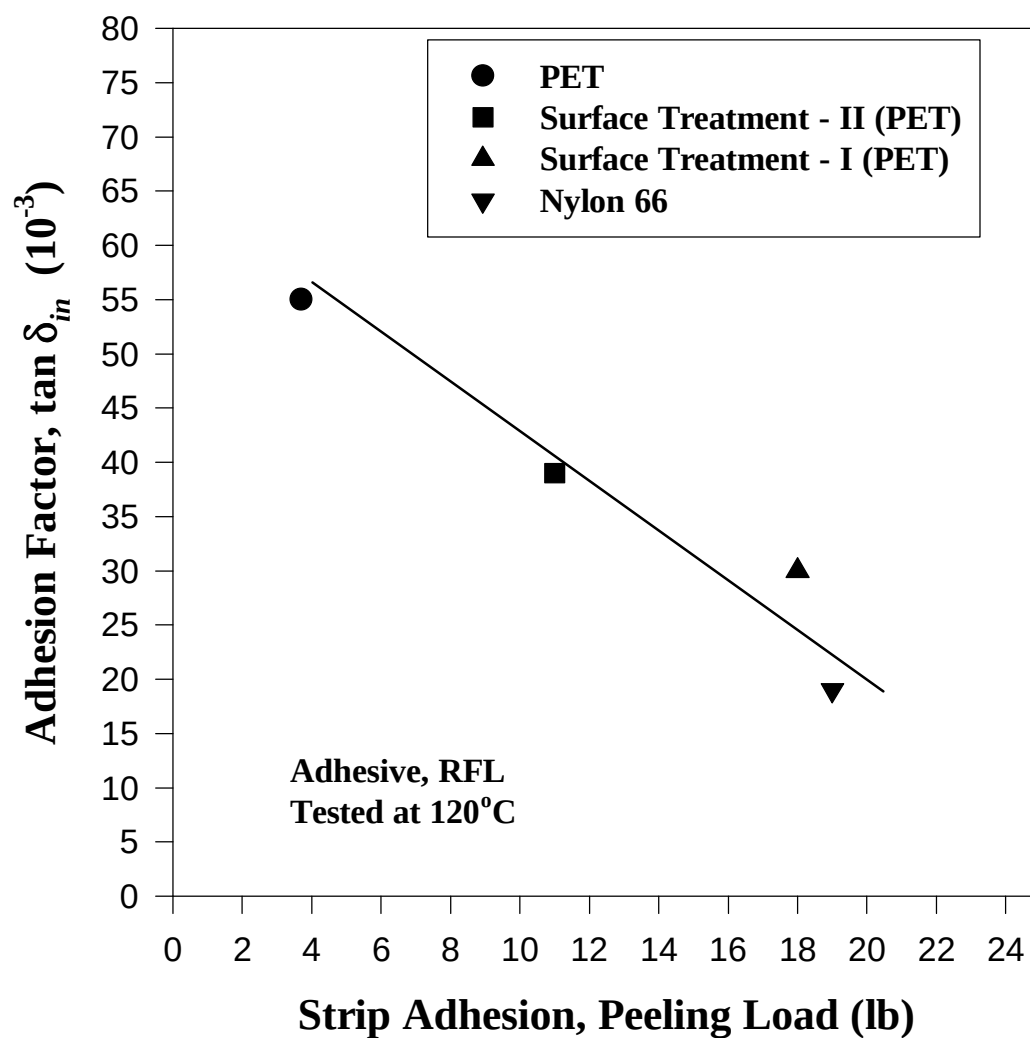


Figure 2.5 Relationship between strip adhesion peeling load and interfacial damping ($\tan \delta_{in}$) from Vibron testing. (adapted from reference [15])

interface of glass fiber polyamide-66 composites, and observed a decrease in damping with the improvement of interfacial bonding. Ko et al. [52] obtained a similar result in the carbon fiber-epoxy system. Contrary to other published work, Chua [53, 54] reported that there was no correlation between interfacial damping measured at room temperature and interfacial adhesion strength for glass fiber-polyester composites. However, he also reported that damping at the glass transition temperature has an inverse relationship with interfacial adhesion strength and transverse flexural strength of composites.

On the basis of the principle that a composite with poor interfacial bonding tends to dissipate more energy than that with good interfacial bonding, Edie et al. [55] applied DMA tests to distinguish carbon fiber-epoxy composites with different fiber surface treatments. The provided results showed much more accuracy than standard mechanical tests.

As mentioned previously, the damping characteristics of unidirectional fiber reinforced composites depend strongly on the orientation of fibers with loading direction. Gerard et al. [56] tested fiber-epoxy composites by using the vibration damping technique. The shear stress was introduced by both torsion and short beam bending. The experiment results proved [56, 57] that the damping property is more interface sensitive for short beam bending because fibers are carrying more of the load in the bending mode. Reed [58] studied glass fiber-epoxy composites and found that in flexural deformation the damping behavior of the composites becomes similar to that of the matrix when the orientation of fibers changed from 0° (corresponding to conventional unidirectional composites) to 90° . This result also proved that interfacial sensitivity depends strongly on the load carrying capability of the fibers; when fibers carry more load, the requirement for interfacial bonding to ensure load transfer is critical. It can be expected that more sensitive detection of the interfacial region in unidirectional composites will be obtained when flexural deformation is applied to the measurements.

In summary, the damping of a composite is largely dependent on interfacial interactions. Damping contributed from the interface, $\tan\delta_{in}$, was proposed to quantify the interfacial bonding in composites although there is no direct quantitative relation between interfacial damping and interfacial adhesion strength in the published work.

2.5. Research Objectives

Based on this review, the main objective of research is to clarify the relationship between dynamic (vibration damping) adhesion measurements and static interfacial properties (interfacial adhesion strength). An optical system was developed to measure the vibration damping of a cantilever beam sample, which is simpler than DMA instrumentation. The sample in the developed system is in the condition of bending deformation and has been proved to significantly reflect interfacial damping of a composite as mentioned previously. A micromechanics model for evaluating the adhesion between fiber and matrix from vibration damping data will be introduced to discuss the experimental results.

CHAPTER 3

EXPERIMENTAL PROCEDURES

Two categories of specimens were used in this study — single fiber and multi-fiber reinforced polymer composites. Single fiber composite specimens were designed for the purpose of direct comparison of the experimental results obtained from microbond pull-out tests and vibration damping experiments. Multi-fiber composite specimens were used as a means to compare these properties to those of composite laminates.

3.1. Sample Preparation

3.1.1. Single-Fiber Composites

As mentioned in Chapter 2, single fiber composite specimens are commonly used to evaluate interfacial adhesion by several static methods such as the pull-out test. A single fiber reinforced composite sample is also preferred in the vibration damping technique because it can be directly compared to single fiber stress analysis of the fiber pull-out test. However, the diameters of fibers should be large enough such that the fiber volume fraction of the single fiber reinforced composite is significant. The resolution of the vibration damping setup used in the experiment is then sufficient to detect the interfacial damping of a single fiber-reinforced composite. Beside glass fibers of 0.12 mm diameter, metallic wires of about 0.1 mm diameter made from copper, molybdenum or tungsten were also used as reinforcement in epoxy matrices as single-fiber composite specimens. The exact diameters of the metallic wires are 0.1 mm for both copper and tungsten, and 0.127 mm for molybdenum. Each metallic wire used here has a unique Young's modulus (copper, molybdenum and tungsten have Young's moduli about 100 GPa, 200 GPa and 300 GPa, respectively), and likely, different adhesion strengths with the epoxy resin. Both vibration damping and microbond specimens were fabricated to measure the interfacial damping and the interfacial adhesion strength.

Sample preparation was conducted according to the procedure suggested by the manufacture of the epoxy resin. Epon 828TM (a trademark of Shell Chemical Company) epoxy resin (100 parts by weight) was mixed with mPDA (1, 3-Phenylenediamine flakes) curing agent

(14.5 parts by weight) when mPDA is completely melted at 70°C. The mixture was placed into a warm vacuum oven (50°C) for 30 minutes to remove gas bubbles introduced during the mixing. The mixture was then used for preparing the single fiber (wire) composite microbond or vibration damping specimens. For the microbond specimens, an axisymmetric drop of epoxy was placed on the fibers (wires) using a small diameter steel needle. For the vibration damping specimens, epoxy was poured into a mold with a size of 30 mm×3 mm×1 mm, into which the single fibers (wires) were placed. In both instances, the prepared specimens were cured for two hours at 75°C and two hours at 125°C. A typical micrograph for single glass fiber-reinforced composite is shown in Figure 3.1.

3.1.2. Multi-Fiber Composites

Laminated multi-fiber composite specimens were fabricated at the Owens Corning Science and Technology Center (Granville, OH). D.E.R. 331 epoxy resin from Dow Chemical Company and Lindride 66 curing agent from Lindau Chemicals Inc. were selected as matrix materials commonly used in the filament winding process. The reinforcement was E-glass fibers with a diameter of 10 μm. Four fiber systems that contained different surface treatments were investigated in this study as listed in Table 3.1. Three fiber volume fractions (about 30%, 50% and 70%) were under control to make the composite laminates.

D.E.R. 331 epoxy (100 parts by weight) was mixed with Lindride 66, curing agent, (85 parts by weight). Composite laminates were fabricated using a filament winding machine and samples were cured for two hours at 120°C and two hours at 180°C using a hot press machine with a 1.43 MPa constant pressure. The composite laminates were then cut into specimens. For the laminates with 30% and 50% fiber volume fractions, the dimension of specimens is about 60 mm in length, 4.5 mm in width with varying thicknesses. The actual lengths of the cantilever beam were varied from 47 mm to 55 mm to obtain an identical resonant frequency. For the laminates with 70% fiber volume fraction, the composite laminates were cut into 30 mm×4 mm×0.5 mm specimens, and the actual length of the cantilever beam was 25 mm. Figure 3.2 shows micrographs of the surface of the E-glass fiber reinforced composite laminates with untreated fiber and different fiber volume fractions.

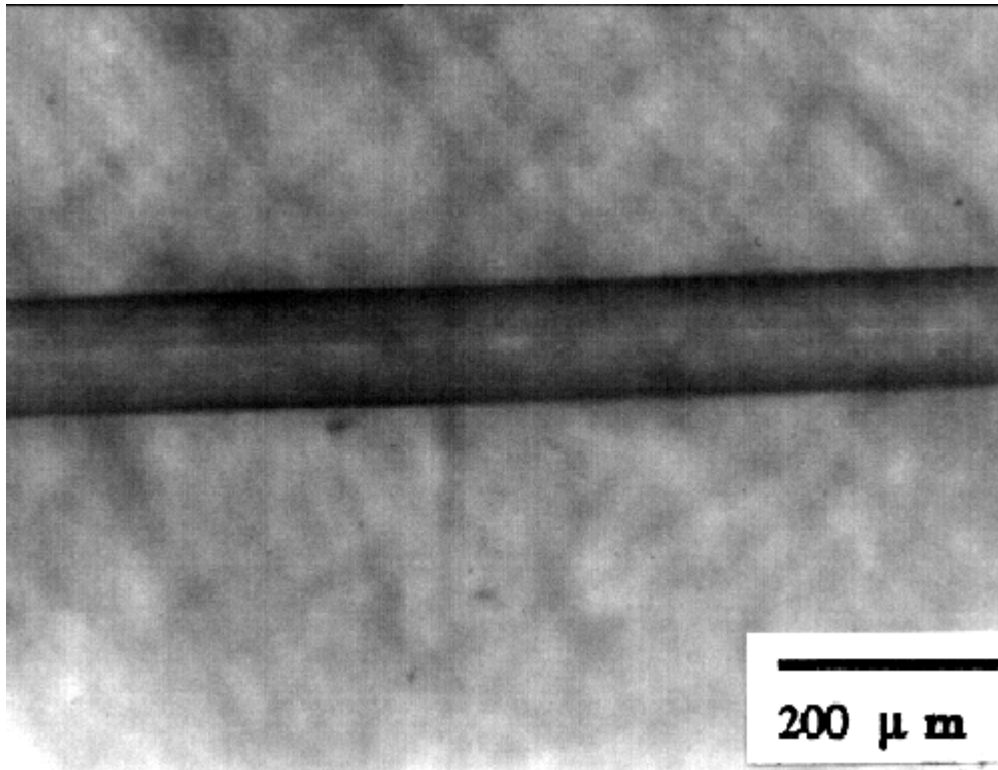
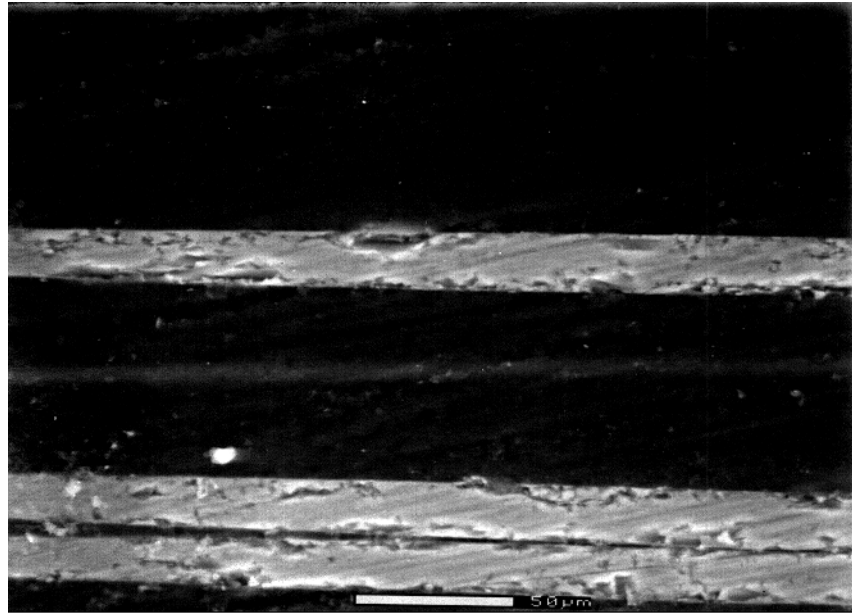


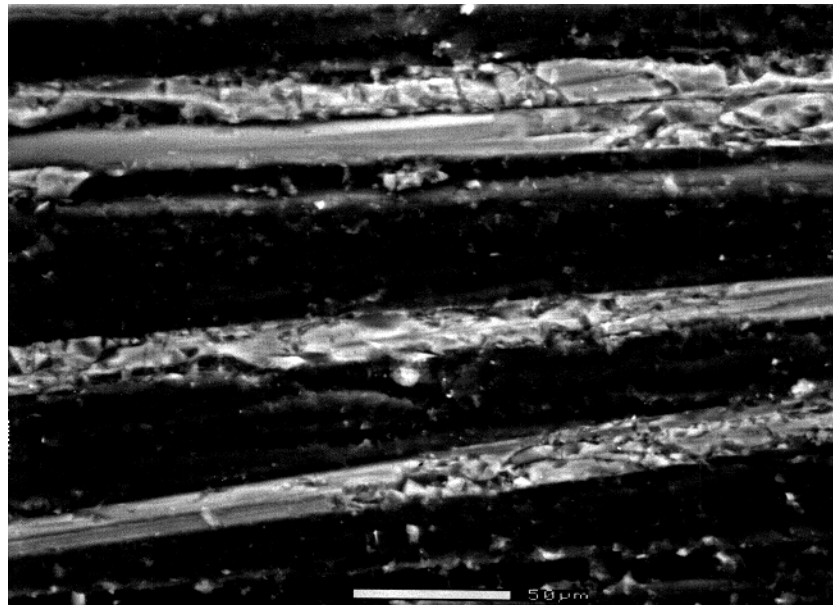
Figure 3.1 Micrograph of single glass fiber-reinforced composite.

Table 3.1 The composite laminate samples used in this study

Specimen Type	Fiber Volume Fraction	Description of Surface Treatment
A	0.325; 0.525; 0.610	Untreated fiber
B	0.332; 0.545; 0.677	366 size without silane
C	0.341; 0.540; 0.695	366 size with silane
D	0.343; 0.515; 0.716	158B size with silane

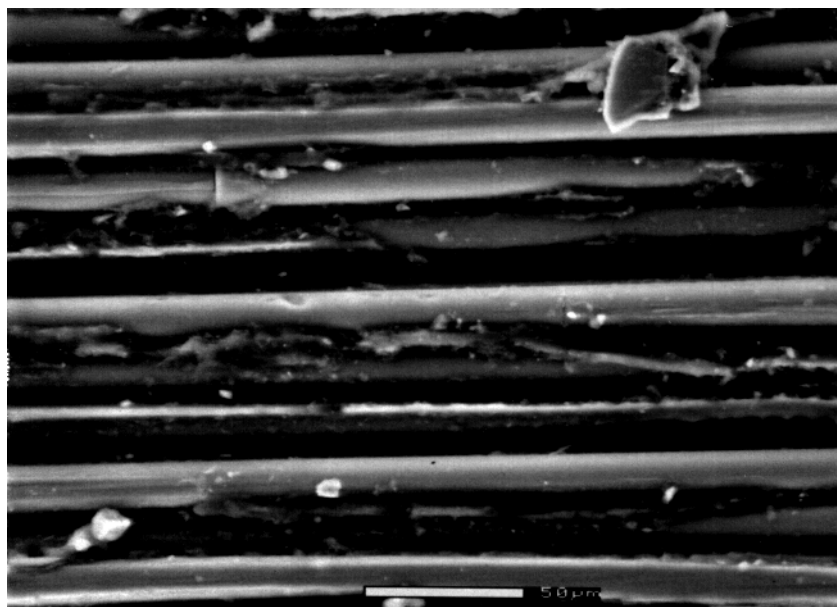


(a)



(b)

Figure 3.2 Micrographs of the E-glass fiber reinforced composite laminates with untreated fiber and different fiber volume fractions (V_f): (a) $V_f = 32.5\%$; (b) $V_f = 52.5\%$; (c) $V_f = 61.0\%$. Bars represent 50 μm . (continued on following page)



(c)

Figure 3.2 Continued from previous page.

In order to verify the adhesion evaluated by vibration damping and static methods, both interfacial adhesion strength measurements and vibration damping tests for a cantilever beam were conducted. For single fiber specimens, microbond pull-out tests were performed to measure the interfacial adhesion strength. For multi-fiber composite specimens, transverse tensile strength measurements were performed at the Owens Corning Science and Technology Center (Granville, OH) to characterize the interfacial adhesion properties.

3.2. Microbond Adhesion Testing of Single-Fiber Composites

Interfacial adhesion strength measurements were made using the microbond fiber pull-out tests. This test was used to measure the force required to debond drops of cured epoxy resin from different fibers (or wires). Figure 3.3 is a schematic diagram of the microbond setup. One end of the fiber (wire) was fixed with adhesive to a hole-punched metal tab which is connected to a balance. The epoxy droplets were sheared off the fiber at a rate of 5 $\mu\text{m}/\text{second}$ using a custom-made microvise. To grip the epoxy droplet, an adjustable micrometer equipped with shearing plates was used. The shearing plates were first brought into contact with both sides of the fiber and then opened slightly to let the fiber, but not the droplet, move between them. A video camera and monitor were used to improve the resolution during this procedure. The microvise was used to translate the fiber and droplet horizontally using a motorized stage. As the shearing plates moved vertically along the fiber, they make full contact with the droplet and an axial force, detected by the balance, is exerted on the droplet. The axial force on the droplet is then transferred to the fiber through a shearing force at the fiber-matrix interface. When the shearing force exceeds the interfacial bond strength, detachment occurs. The data was transmitted from the balance to a computer at a speed of 6 force readings/second. The maximum force read from the computer is taken as the point at which the droplet has debonded from the fiber. Equation (2.1) was used to calculate interfacial adhesion strength.

3.3. Transverse Tensile Strength Measurements of Multi-Fiber Composites

Transverse tensile strength is a simple and quick alternative to qualitatively evaluate the

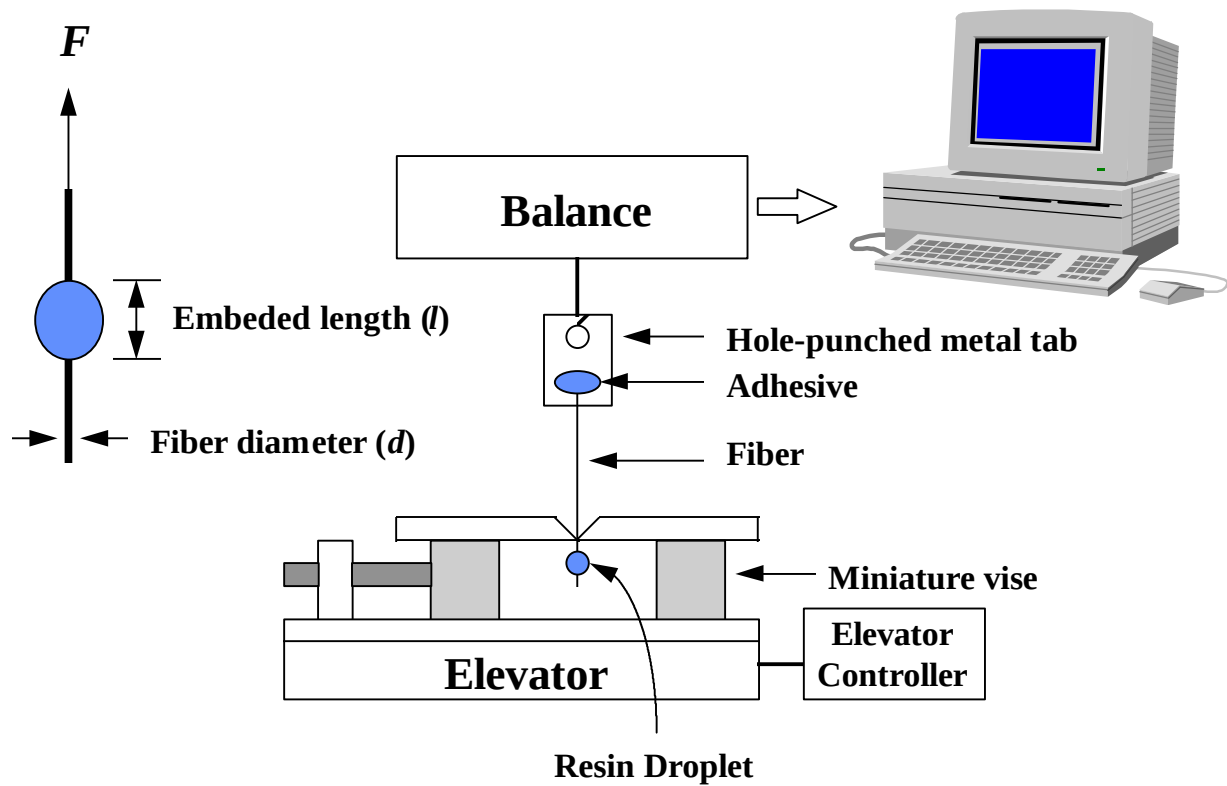


Figure 3.3 Schematic diagram of the microbond single fiber pull-out test. (adapted from reference [59])

fiber-matrix interfacial adhesion. The experiments were conducted by strictly following ASTM test method D3039/D3039M. In this case, a thin flat strip of the composite laminate specimen having a constant rectangular cross-section was mounted in the grips of a universal testing machine (Instron model 1137) and monotonically loaded in tension with the loading direction perpendicular to the fiber longitudinal direction. The load was recorded by the load cell of the testing machine. The ultimate tensile strength of the material was determined from dividing the maximum load carried prior to failure by the cross-sectional area. It is obvious that stronger interface provides a higher transverse tensile strength as long as the observed failure occurs at the fiber-matrix interface. If matrix failure is found, the data will not reflect the nature of the interface and are not valid.

3.4. Optical Measurements of Vibration Damping

A schematic of the optical setup designed to measure the deflection and vibration dynamics of a cantilever beam is shown in Figure 3.4. The apparatus consists of a 1 mW solid state laser (670 nm), a mirror, a beam splitter and a position sensitive photodetector. A sample is mounted by clamping it vertically between two plates such that the protruding length forms a cantilever beam. An electronically triggered pin is used to generate an initial deflection on the sample; vibration of the sample is initiated by retracting the pin. Vibration curves are obtained by reflecting the laser beam off the sample to the photodetector.

It is necessary to emphasize the importance of the clamping apparatus. This fixture was designed to minimize sources of energy dissipation. Two pieces of aluminum plate were used to form a clamp and two pairs of bolts and nuts were utilized to apply a uniform clamping pressure. The overall consistency of the vibration damping curves from the same specimen and the small deviation of the results among different specimens in the same condition are the evidence of successful design of the clamp.

The damping factor, $\tan\delta$, is calculated from the decaying-oscillatory damping curve by the following [29]

$$\tan\delta = \frac{\ln(A_0 / A_n)}{n\pi} \quad (3.1)$$

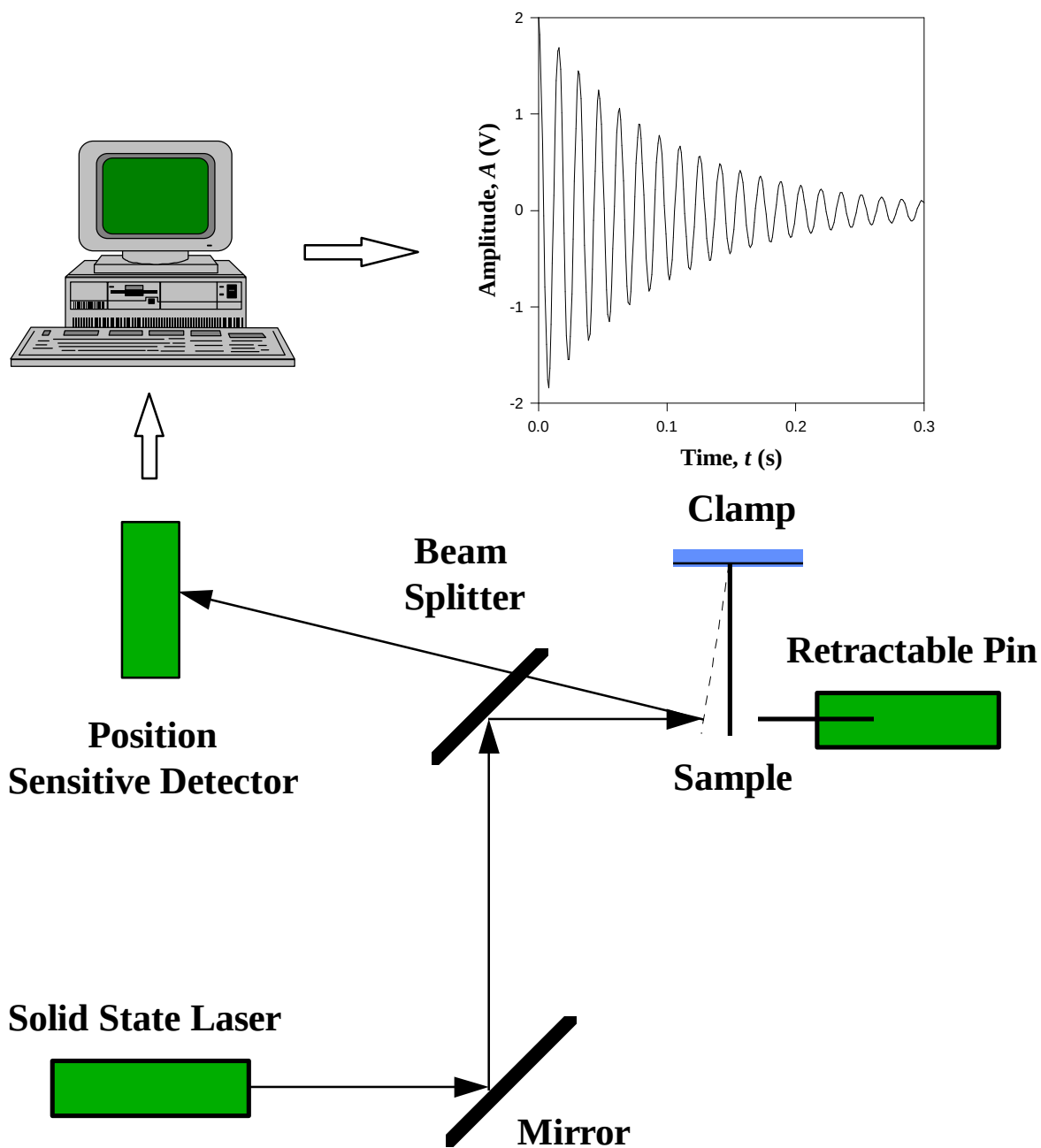


Figure 3.4 Schematic diagram of the optical system.

where n is the number of cycles of the vibration, A_0 is the amplitude of the first vibration, and A_n is amplitude of the n^{th} vibration. The term $\frac{\ln(A_0 / A_n)}{n}$, also known as the logarithmic decrement d , is proportional to another damping factor ζ (Equation (2.15)). It can be obtained by fitting the experimental data to the following equation [29] to determine ζ :

$$A(t) = B_0 \exp(-\zeta \omega_r t) \cos(\omega_d t - \phi) + B_1 \quad (3.2)$$

where B_0 , and B_1 are constants.

Measurements of vibration damping are complicated by the fact that $\tan \delta$ usually is a function of the amplitude of the vibration. However, it appears that for small amplitudes, $\tan \delta$ is reasonably constant [27].

CHAPTER 4

EXPERIMENTAL RESULTS

The interfacial adhesion was evaluated for both single-fiber and multi-fiber reinforced polymer composites with different techniques, i.e., microbond pull-out tests, transverse tensile strength measurements and vibration damping techniques. The experimental results are summarized as follows. Note that the values presented in all of the tables are the average measurements from at least five specimens unless otherwise specified.

4.1. Single-Fiber Composites

The interfacial adhesion strength measurements obtained from the microbond single fiber (wire) pull-out tests are summarized in Table 4.1. For all of the metallic wire reinforced polymer composites used in the current study, the wires were not only supplied in as-received condition, but also coated with silicone mold release to create a weak wire-matrix interface. The measurements show clearly that all silicone mold release coated wires exhibit lower interfacial adhesion strengths. The experimental values listed in Table 4.1 are averages obtained from at least twenty microbond specimens.

Figure 4.1 shows a typical example of the vibration damping curve of a single glass fiber reinforced composite sample, which was obtained from the optical system described in Section 3.4. The value of $\tan \delta$ was found to be 39.8×10^{-3} which is typical for this material system at the fixed resonant frequency. Other experimental results are summarized in Table 4.2.

4.2. Multi-Fiber Composites

Data in Table 4.3 represent the transverse tensile strength of the composite laminates used in the current study. Figure 4.2 is the similar curve as Figure 4.1, but was obtained from a composite laminate specimen with 61% volume fraction of non-surface-treated E-glass fibers (designated as Type A specimen). The value of $\tan \delta$ was found to be 3.81×10^{-3} which is also

Table 4.1 The interfacial adhesion strength obtained from the microbond single fiber pull-out tests

Fibers (Wires)	Fiber Diameter (μm)	Interfacial Adhesion Strength, τ_{measure} (KPa)
Mo	127	1071 (± 315) *
Mo (with silicone mold release)	127	788 (± 396)
Cu	100	1263 (± 324)
Cu (with silicone mold release)	100	830 (± 317)
W	100	1296 (± 498)
W (with silicone mold release)	100	569 (± 393)
Glass	120	329 (± 207)

*Numbers in parenthesis represent ± 1 standard deviation.

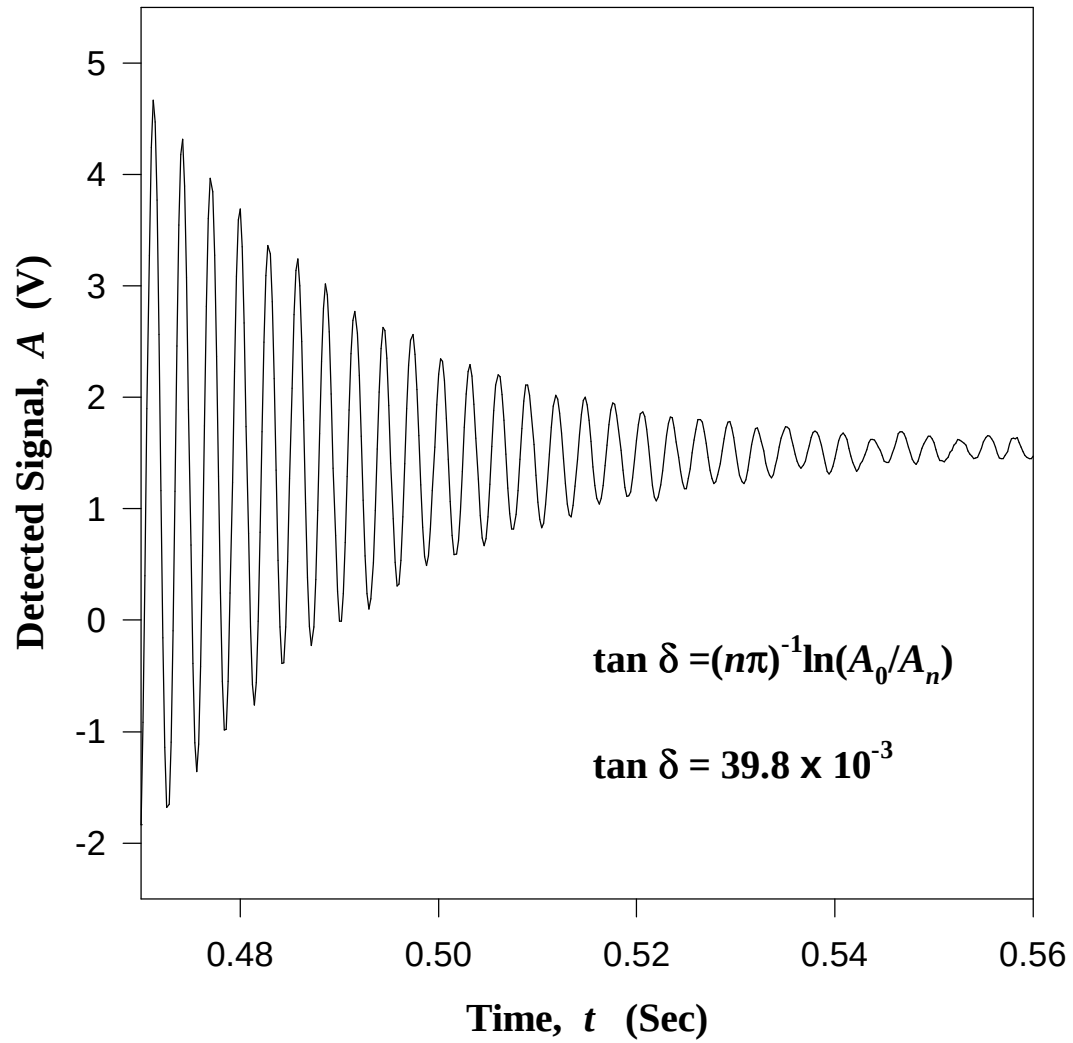


Figure 4.1 An example of the vibration damping curves obtained from single glass fiber reinforced composite samples using the optical system.

Table 4.2 Measured resonant frequencies and damping factors of single fiber (wire) reinforced composites

Fibers	Width, b (mm)	Thickness, h (mm)	ω_r (s^{-1})	$\tan \delta_c$ (10^{-3})
Mo	2.871 (± 0.117)**	0.991 (± 0.040)	2754 (± 115)	43.1 (± 2.78)
Mo*	2.898 (± 0.106)	0.960 (± 0.039)	2759 (± 133)	40.6 (± 3.78)
Cu	2.897 (± 0.111)	0.908 (± 0.046)	2897 (± 137)	45.5 (± 1.56)
Cu*	2.993 (± 0.050)	0.923 (± 0.036)	2902 (± 169)	47.4 (± 1.62)
W	2.912 (± 0.060)	0.798 (± 0.043)	2514 (± 130)	41.9(± 2.15)
W*	2.851 (± 0.068)	0.838 (± 0.036)	2829 (± 93)	37.7(± 4.29)
Glass	2.858 (± 0.096)	0.857 (± 0.075)	2427 (± 195)	39.8 (± 1.06)

* With silicone mold release.

**Numbers in parenthesis represent ± 1 standard deviation.

Table 4.3 Transverse tensile strength of composite laminates

Specimen Type	Fiber Volume Fraction	Width, b (mm)	Thickness, h (mm)	σ_{tr} (MPa)
A	0.325	4.61 (± 0.37)*	1.72 (± 0.01)	19 (± 3)
	0.525	4.65 (± 0.28)	1.59 (± 0.01)	19 (± 4)
	0.610	4.02 (± 0.05)	0.51 (± 0.01)	28 (± 4)
B	0.332	4.38 (± 0.29)	1.34 (± 0.01)	20 (± 10)
	0.545	4.66 (± 0.19)	1.51 (± 0.02)	33 (± 5)
	0.677	4.08 (± 0.02)	0.51 (± 0.01)	45 (± 4)
C	0.341	4.39 (± 0.21)	1.54 (± 0.02)	21 (± 5)
	0.540	4.65 (± 0.24)	1.54 (± 0.01)	39 (± 6)
	0.695	3.99 (± 0.05)	0.51 (± 0.01)	56 (± 5)
D	0.343	4.63 (± 0.25)	1.54 (± 0.04)	19 (± 3)
	0.515	4.65 (± 0.15)	1.50 (± 0.00)	20 (± 2)
	0.716	4.05 (± 0.05)	0.51 (± 0.00)	46 (± 4)

*Numbers in parenthesis represent ± 1 standard deviation.

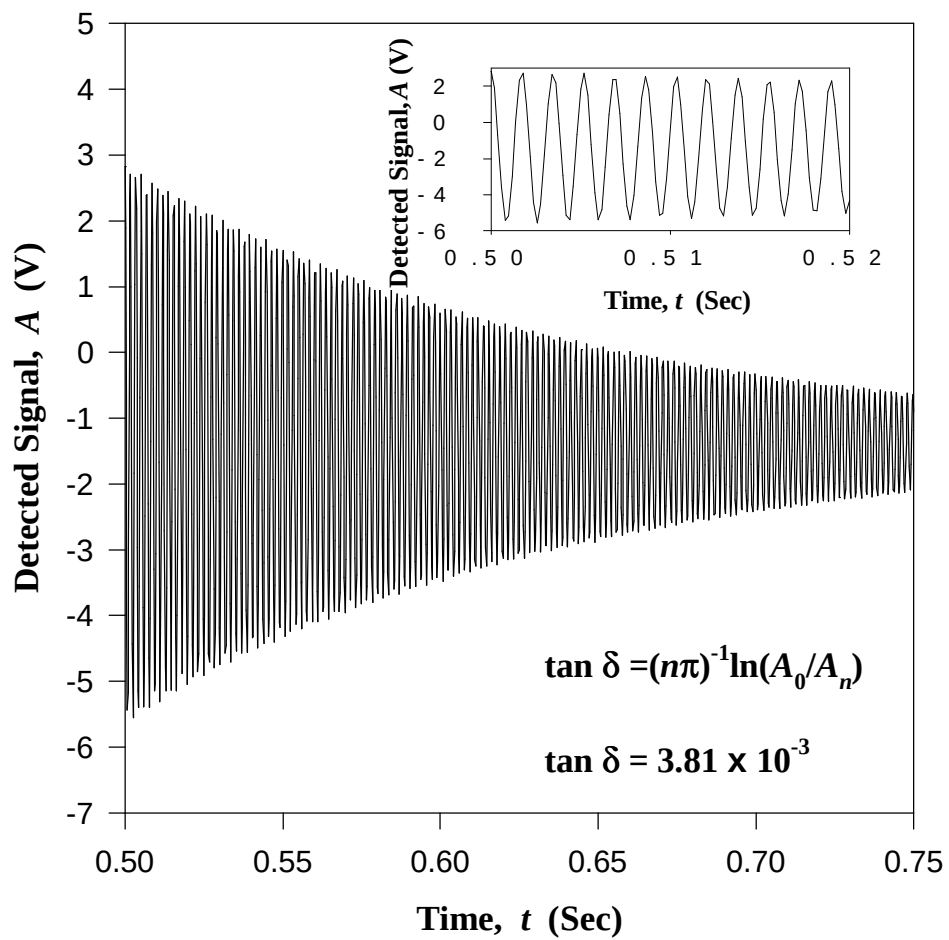


Figure 4.2 An example of the vibration damping curves from composite laminate specimens measured using the optical system.

typical for this material system at the fixed resonant frequency. The rest of data are listed in Table 4.4.

It is important to note from Tables 4.1 through 4.4 that the overall vibration damping characteristics and resonant frequencies of the tested specimens depend on sample dimensions. It can be seen that there is no obvious relationship between the overall damping factor of the composites and the interfacial adhesion strength. Therefore, it is necessary to separate the contribution of energy dissipation from the component materials and the interface to determine the correspondence of the interfacial damping and interfacial adhesion strength. As mentioned in Chapter 2, Equation (2.18) can be used for this purpose. However, prior to applying Equation (2.18) to calculate the interfacial damping contribution, it is necessary to know the values of certain parameters including the Young's modulus of the composites, and the Young's moduli and $\tan \delta$ for each component. In the current study, some values were obtained by experiment, while some data were obtained from the literature.

From the Bernoulli- Euler beam equation [29, 60, 61], it can be shown that the Young's modulus, E , of the material is related to its frequency of vibration. The equation used for calculating E for a beam specimen is as follows [29]:

$$E = \frac{12\rho\omega_r^2 L^4}{1.875^4 h^2} \quad (4.1)$$

where ω_r is the resonant frequency of the first mode of vibration, L and h are the length and the thickness of the beam, and ρ is the beam density. The density of the epoxy resin is reported from 1.115 g/cm³ [62] to 1.25 g/cm³ [63]. In the current study, the former value was used for the Epon 828 epoxy resin which was used to make single fiber (wire) reinforced epoxy composite specimens. For the E-glass fiber reinforced epoxy composite laminates provided by Owens Corning Science & Technology Center (Granville, OH), the latter value was used for the epoxy density. Rule of mixtures (ROM) was utilized to obtain the beam density for all composite beams.

It has been reported that the damping factor also varies with frequency [48, 64]. By changing the beam length, an identical natural frequency (or resonant frequency) was obtained for the single component materials, which was comparable to the frequencies of the tested composites. The Young's moduli and damping factors measured from the optical vibration

**Table 4.4 Measured resonant frequencies and damping factors
of composite laminate specimens**

Specimen Type	Fiber Volume Fraction	Width, b (mm)	Thickness, h (mm)	ω_r (s ⁻¹)	$\tan \delta_c$ (10 ⁻³)
A	0.325	4.61 (±0.37)*	1.72 (±0.01)	2062 (±110)	5.13 (±0.37)
	0.525	4.65 (±0.28)	1.59 (±0.01)	2129 (±46)	3.72 (±0.32)
	0.610	4.02 (±0.05)	0.51 (±0.01)	3733 (±74)	3.74 (±0.06)
B	0.332	4.38 (±0.29)	1.34 (±0.01)	2100 (±48)	4.11 (±0.15)
	0.545	4.66 (±0.19)	1.51 (±0.02)	2106 (±37)	3.32 (±0.17)
	0.677	4.08 (±0.02)	0.51 (±0.01)	3976 (±106)	2.75 (±0.10)
C	0.341	4.39 (±0.21)	1.54 (±0.02)	2058 (±50)	4.25 (±0.13)
	0.540	4.65 (±0.24)	1.54 (±0.01)	2111 (±27)	3.14 (±0.12)
	0.695	3.99 (±0.05)	0.51 (±0.01)	4061 (±50)	2.46 (±0.12)
D	0.343	4.63 (±0.25)	1.54 (±0.04)	2112 (±82)	4.07 (±0.15)
	0.515	4.65 (±0.15)	1.50 (±0.00)	2098 (±35)	3.39 (±0.32)
	0.716	4.05 (±0.05)	0.51 (±0.00)	4038(±47)	2.73 (±0.02)

*Numbers in parenthesis represent ±1 standard deviation.

damping system are summarized in Tables 4.5 and 4.6. Note that the data for single glass fiber were borrowed from the E-glass values [48] which have been reported not to change much for various kinds of glasses. The average values of the data listed in Tables 4.5 and 4.6 were used to calculate $\tan \delta_{in}$ based on Equation (2.18). The results are given in Tables 4.7 and 4.8.

The variation of interfacial damping factor $\tan \delta_{in}$ with the change of volume fraction of E-glass fibers V_f is plotted in Figure 4.3. It is evident that $\tan \delta_{in}$ increases with increasing of V_f for each type of E-glass fiber reinforced epoxy composite laminate. As the volume fraction of glass fibers in the composites increase, more fiber-matrix interfacial area is created, and the more energy can be dissipated by the fiber-matrix interface.

When comparing values of $\tan \delta_{in}$ listed in Table 4.7 with the measured interfacial adhesion strength $\tau_{measure}$ shown in Table 4.1, it can be recognized immediately that there is an inverse relationship between the two parameters (refer to Figure 4.4).

It is shown that the transverse tensile strength σ_{tr} is usually proportional to τ [65]. The transverse tensile strength σ_{tr} thus can be used to qualitatively evaluate the fiber-matrix interfacial adhesion. Figure 4.5 through 4.7 are the comparison of $\tan \delta_{in}$ and σ_{tr} of the composite laminates with different volume fractions of E-glass fibers. As an example, Figure 4.8 shows the fracture surfaces for all the types of glass-epoxy composite laminates with approximately 70% volume fraction of E-glass fibers.

Table 4.5 Densities, Young's moduli and damping factors of component materials of single fiber (wire) reinforced composites

	ρ (g/cm ³)	E (GPa)	$\tan \delta$ (10 ⁻³)
Epon 828 epoxy resin	1.115 ^①	3.0 (± 0.2) ^②	43.1(± 1.8)
Copper	8.93 ^③	98 (± 7)	3.20 (± 0.26)
Molybdenum	10.22 ^③	220 (± 11)	3.20 (± 0.12)
Tungsten	19.3 ^③	315 (± 16)	2.90 (± 0.19)
Glass	2.54 ^④	60 ^⑤	1.0 ^⑤

^① Obtained from reference [62].

^② Numbers in parenthesis represent ±1 standard deviation.

^③ Products of Alfa[®] Aesar[®].

^④ Obtained from reference [48].

^⑤ Provided by Owens Corning Science & Technology Center.

Table 4.6 Young's moduli and damping factors of component materials of E-glass fiber reinforced composite laminates

	ρ (g/cm ³)	E (GPa)		$\tan \delta$ (10 ⁻³)	
Epoxy resin	1.25 ^①	2.43 (± 0.03) ^{②③}	3.21 (± 0.05) ^④	25.2 (± 0.3) ^⑤	21.8 (± 0.3) ^④
E-Glass	2.54 ^⑥	60 ^①		1.0 ^⑥	

^① Provided by Owens Corning Science & Technology Center.

^② Numbers in parenthesis represent ± 1 standard deviation.

^③ For samples with about 70% volume fraction of glass fibers.

^④ For samples with about 30% and 50% volume fractions of glass fibers.

^⑥ Obtained from reference [48].

**Table 4.7 Damping factors contributed from interface of single fiber (wire)
reinforced composites**

Fibers	Width, b (mm)	Thickness, h (mm)	$\tan \delta_{in}$ (10^{-3})
Mo	2.871 (± 0.117)**	0.991 (± 0.040)	0.58 (± 0.27)
Mo*	2.898 (± 0.106)	0.960 (± 0.039)	0.79 (± 0.30)
Cu	2.897 (± 0.111)	0.908 (± 0.046)	0.58 (± 0.18)
Cu*	2.993 (± 0.050)	0.923 (± 0.036)	1.29 (± 0.51)
W	2.912 (± 0.060)	0.798 (± 0.043)	0.34 (± 0.13)
W*	2.851 (± 0.068)	0.838 (± 0.036)	1.67 (± 0.76)
Glass	2.858 (± 0.096)	0.857 (± 0.075)	0.80 (± 0.21)

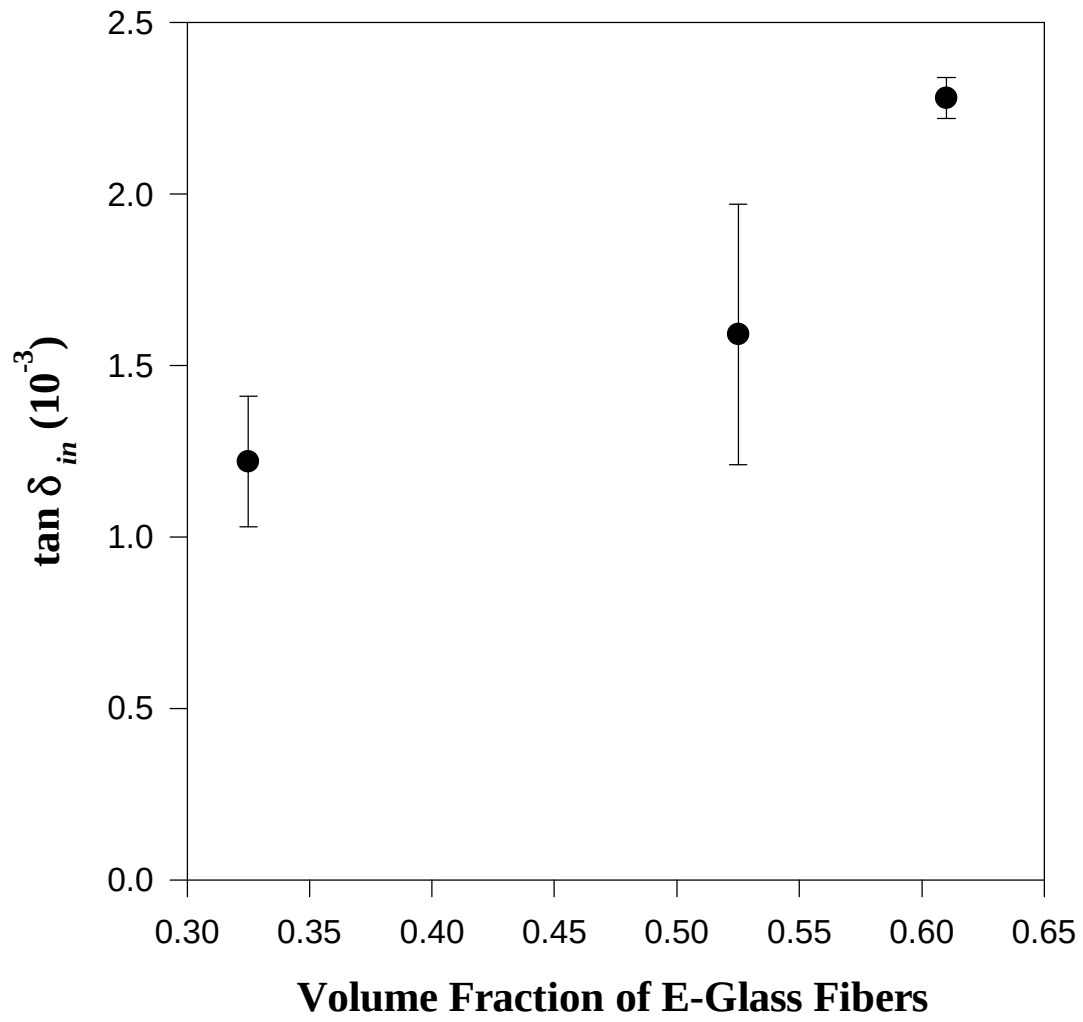
* With silicone mold release.

**Numbers in parenthesis represent ± 1 standard deviation.

**Table 4.8 Damping factors contributed from interface
of composite laminate specimens**

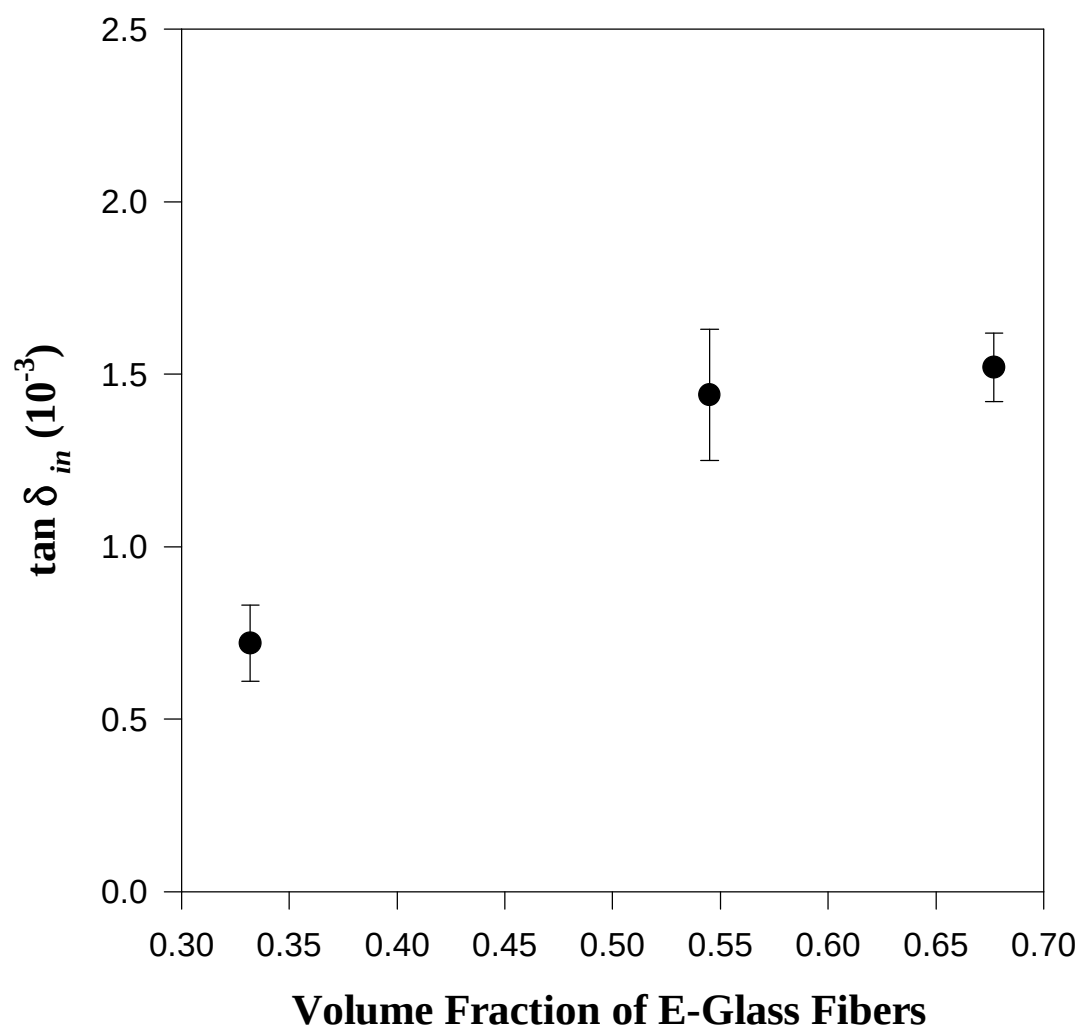
Specimen Type	Fiber Volume Fraction	Width, b (mm)	Thickness, h (mm)	$\tan \delta_{in} (10^{-3})$
A	0.325	4.61 (± 0.37)*	1.72 (± 0.01)	1.22 (± 0.19)
	0.525	4.65 (± 0.28)	1.59 (± 0.01)	1.59 (± 0.38)
	0.610	4.02 (± 0.05)	0.51 (± 0.01)	2.28 (± 0.06)
B	0.332	4.38 (± 0.29)	1.34 (± 0.01)	0.72 (± 0.11)
	0.545	4.66 (± 0.19)	1.51 (± 0.02)	1.44 (± 0.19)
	0.677	4.08 (± 0.02)	0.51 (± 0.01)	1.52 (± 0.10)
C	0.341	4.39 (± 0.21)	1.54 (± 0.02)	0.64 (± 0.08)
	0.540	4.65 (± 0.24)	1.54 (± 0.01)	1.14 (± 0.14)
	0.695	3.99 (± 0.05)	0.51 (± 0.01)	1.28 (± 0.12)
D	0.343	4.63 (± 0.25)	1.54 (± 0.04)	0.79 (± 0.08)
	0.515	4.65 (± 0.15)	1.50 (± 0.00)	1.47 (± 0.38)
	0.716	4.05 (± 0.05)	0.51 (± 0.00)	1.67 (± 0.02)

*Numbers in parenthesis represent ± 1 standard deviation.



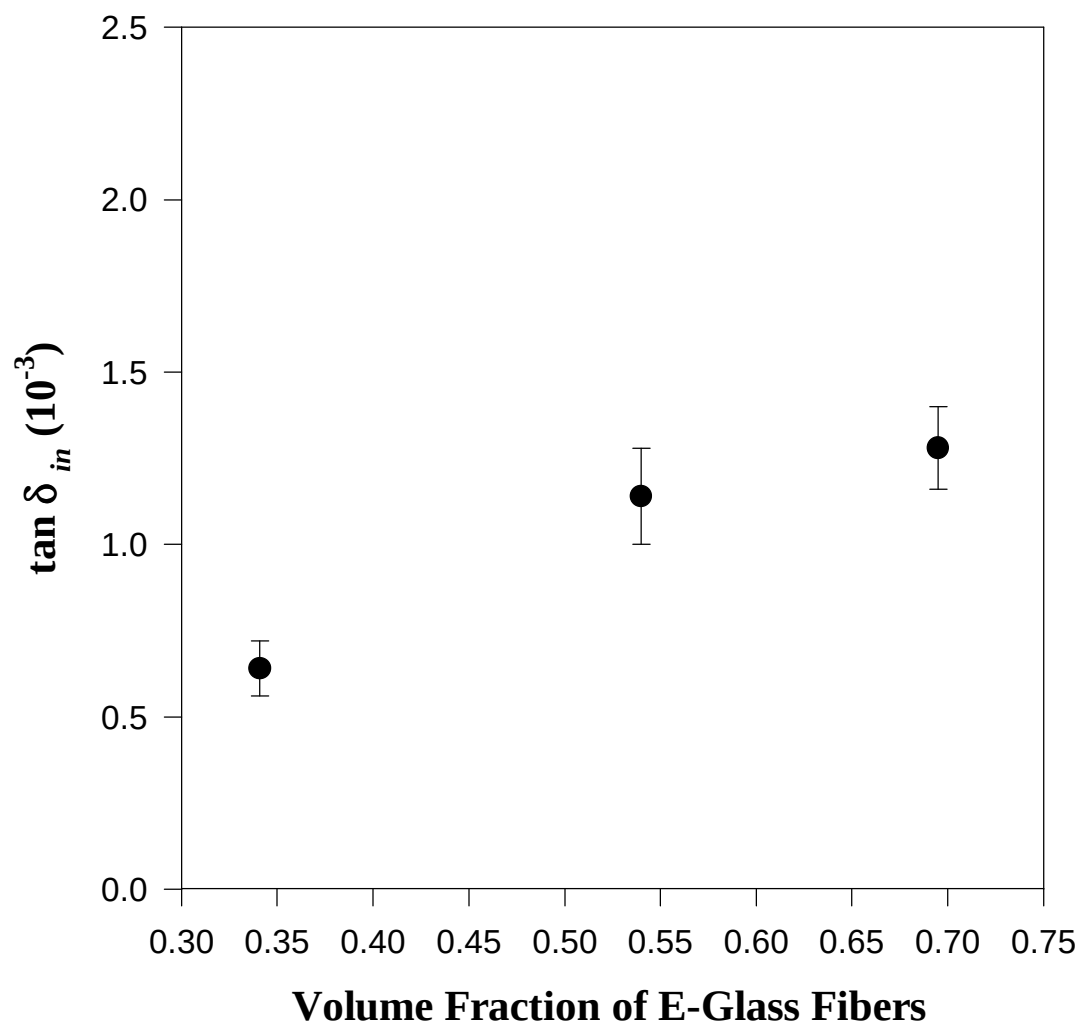
(a)

Figure 4.3 $\tan \delta_{in}$ vs. V_f for different types of E-glass fiber reinforced epoxy composite laminates: (a) Type A specimens; (b) Type B specimens; (c) Type C specimens; (d) Type D specimens. (continued on following pages)



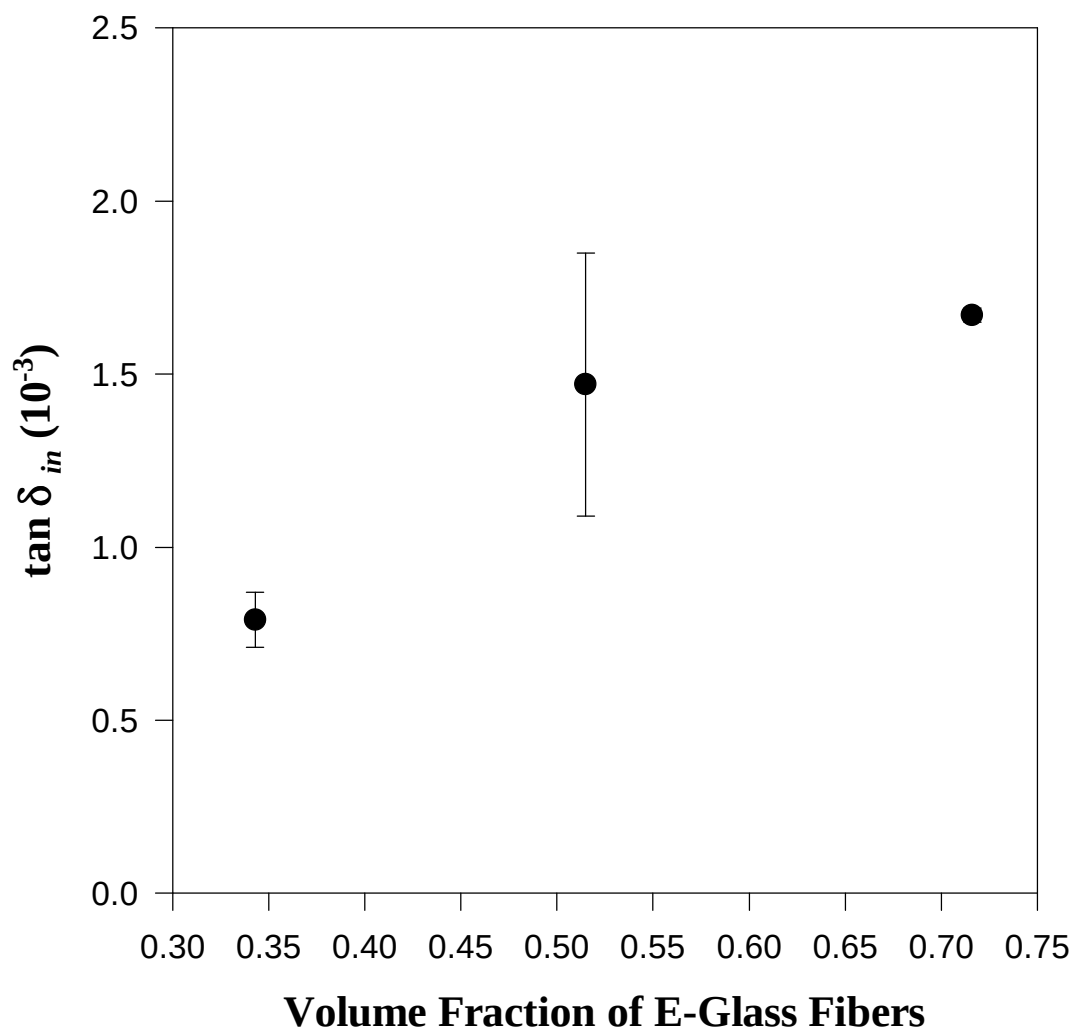
(b)

Figure 4.3 Continued from previous page.



(c)

Figure 4.3 Continued from previous page.



(d)

Figure 4.3 Continued from previous page.

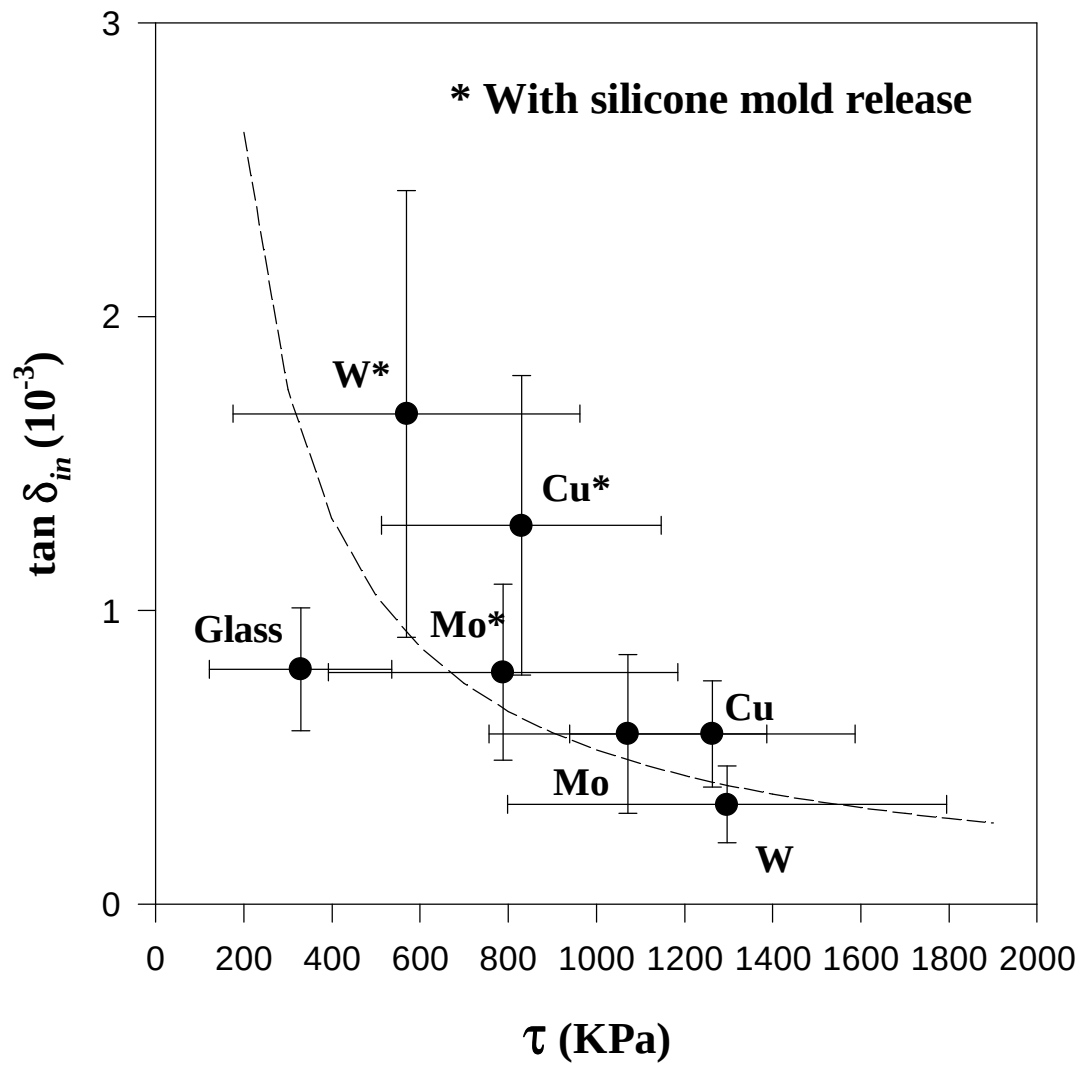


Figure 4.4 The relationship between $\tan \delta_{in}$ and interfacial adhesion strength for single fiber (wire) reinforced composites.

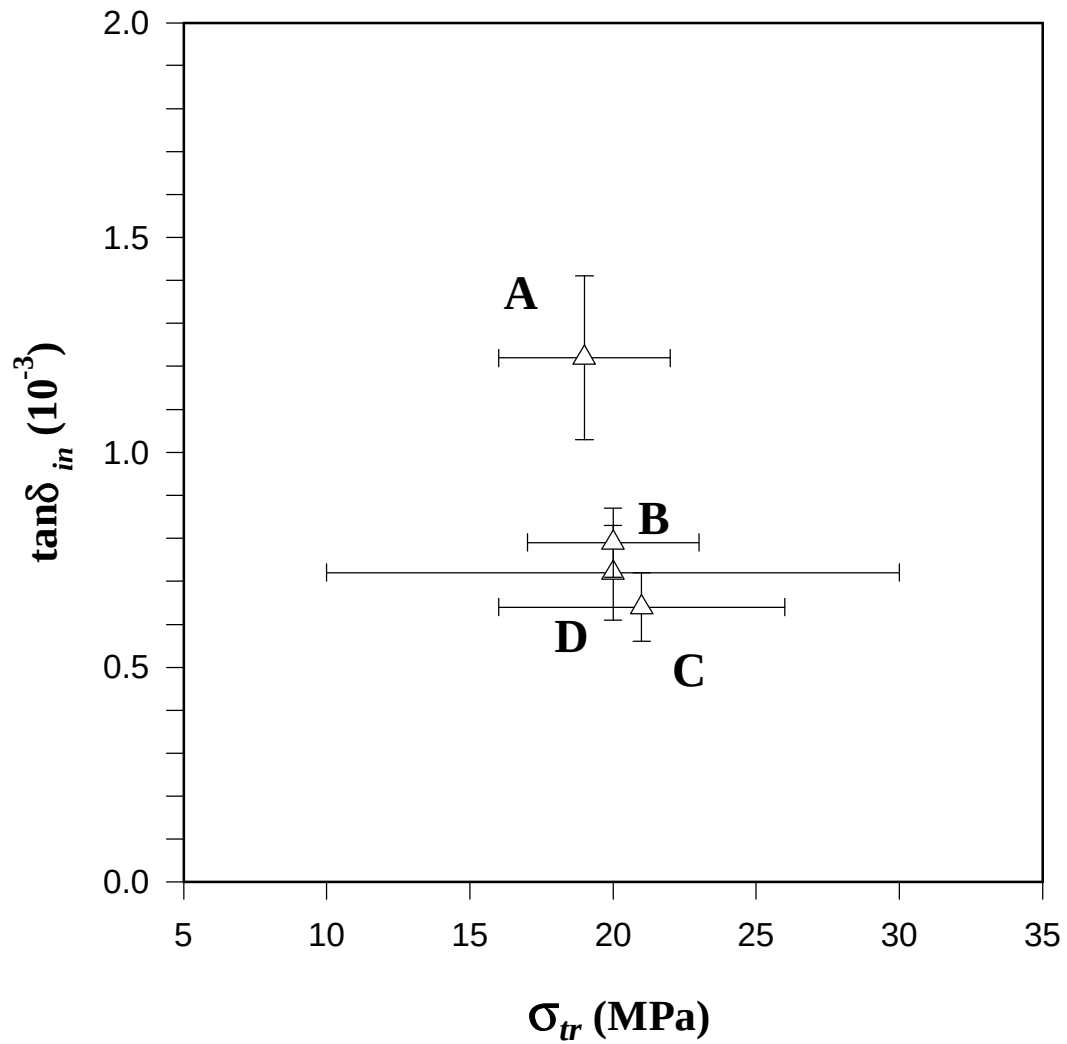


Figure 4.5 The relationship between $\tan \delta_{in}$ and σ_{tr} for composite laminates with about 30% glass fiber volume fraction. Error bars represent ± 1 standard deviation.

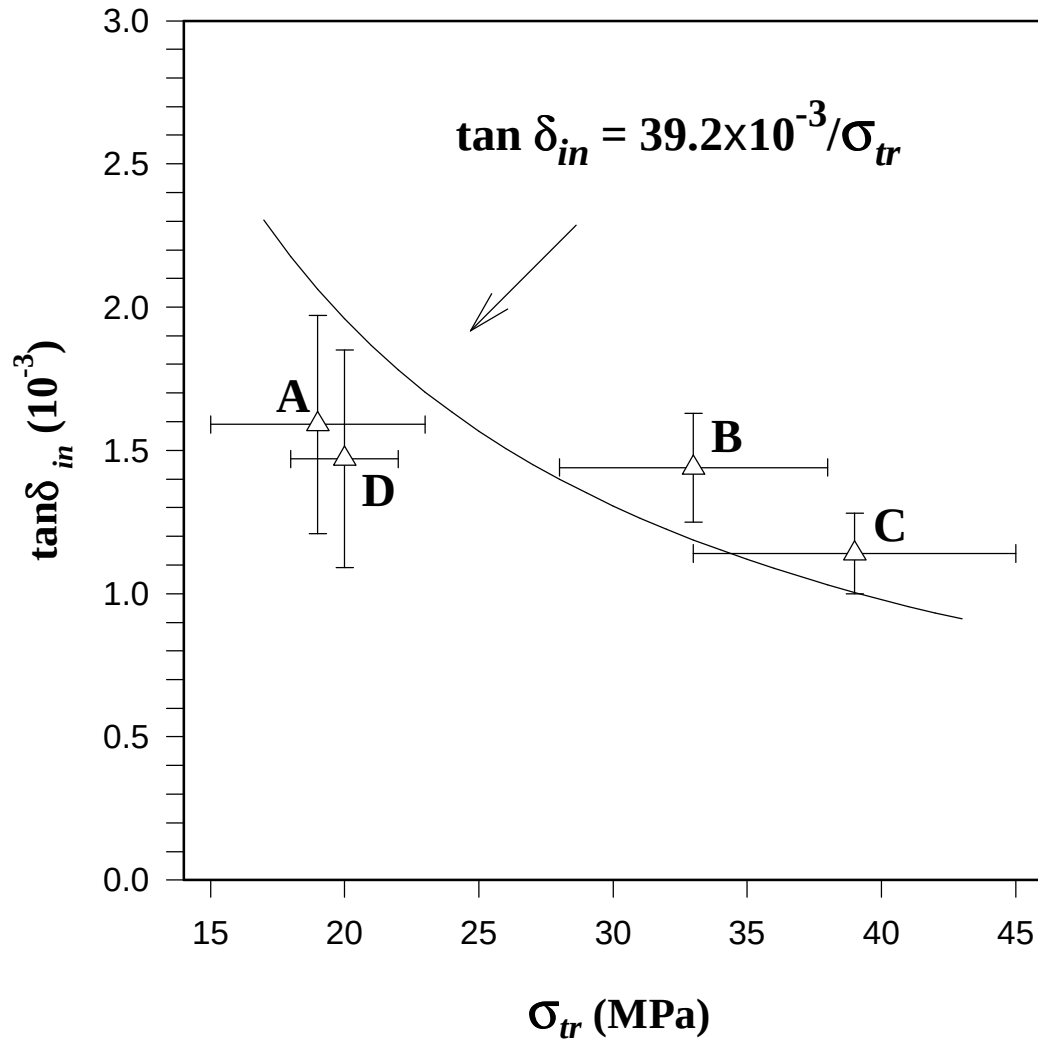


Figure 4.6 The relationship between $\tan \delta_{in}$ and σ_{tr} for composite laminates with about 50% glass fiber volume fraction. Error bars represent ± 1 standard deviation.

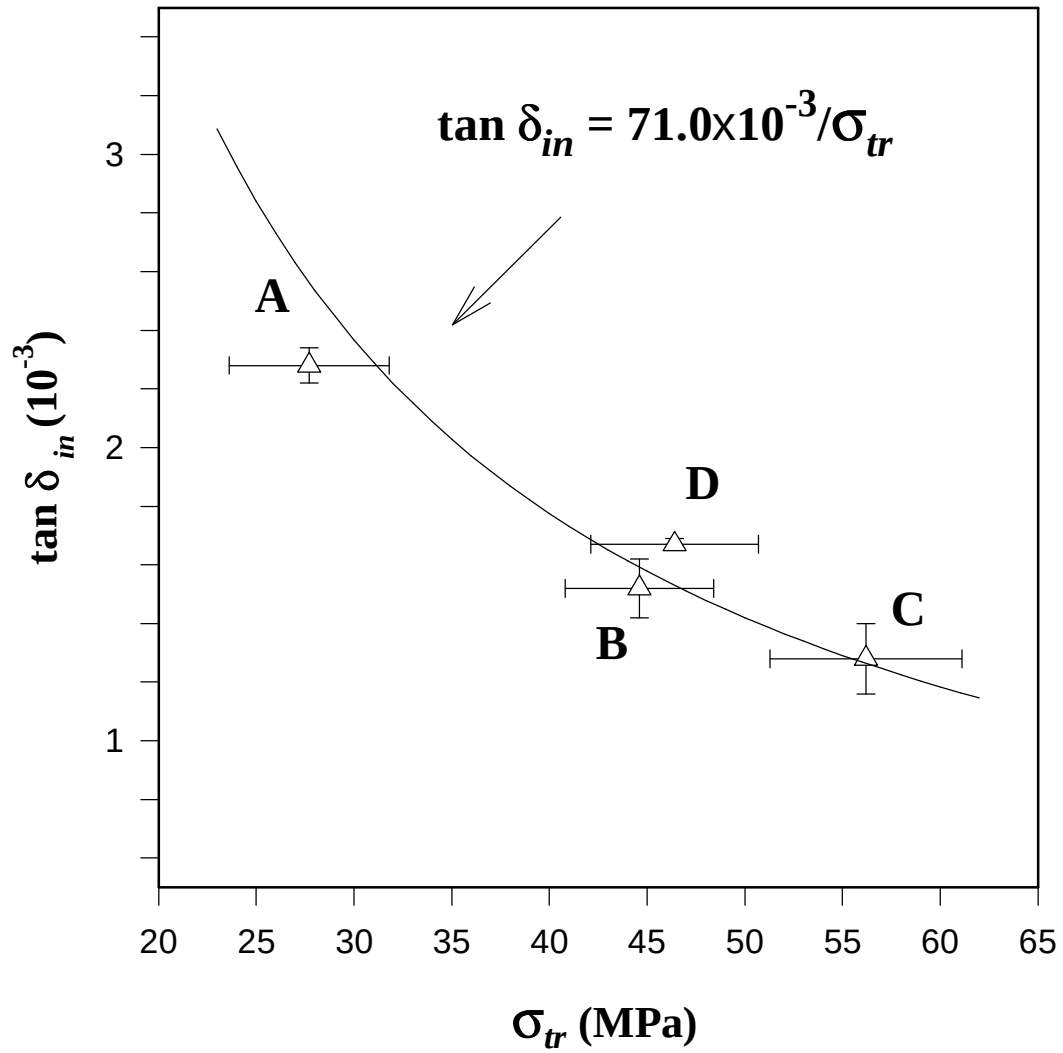
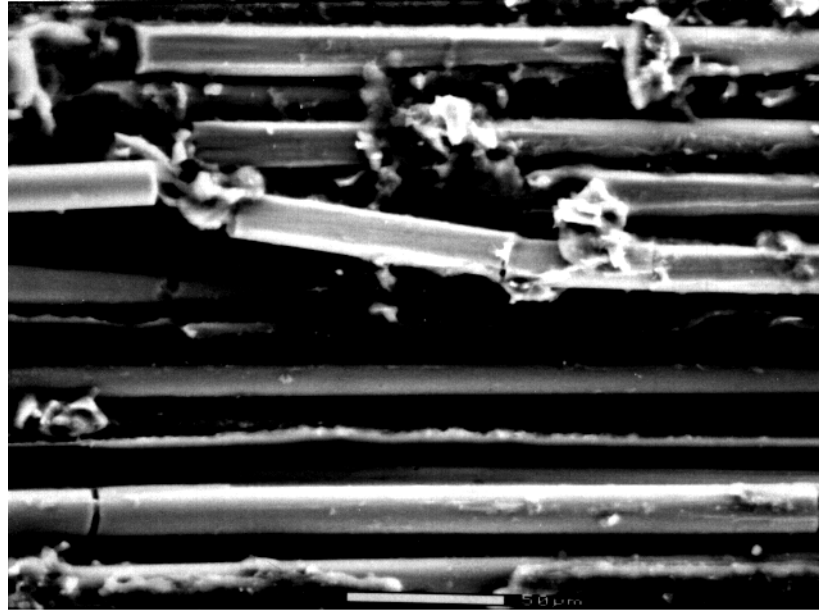
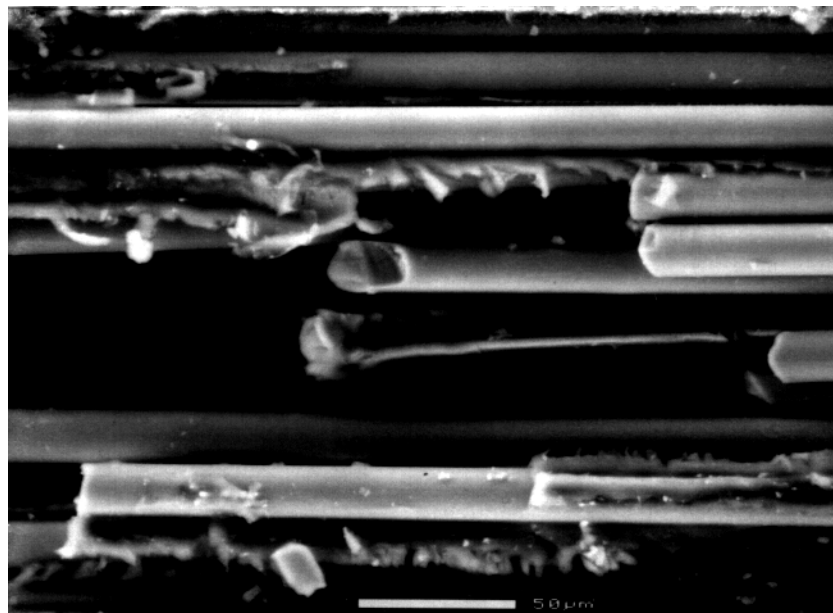


Figure 4.7 The relationship between $\tan \delta_{in}$ and σ_{tr} for composite laminates with about 70% glass fiber volume fraction. Error bars represent ± 1 standard deviation.

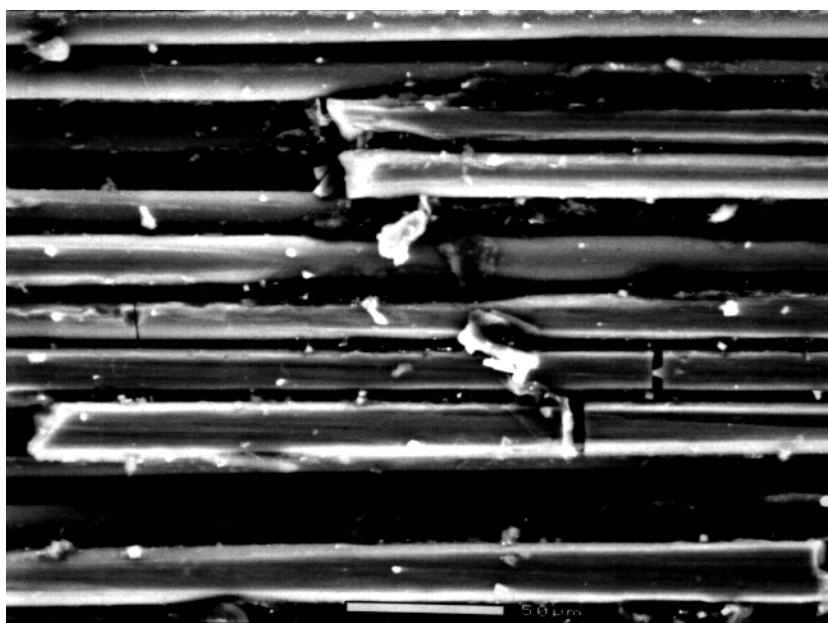


(a)

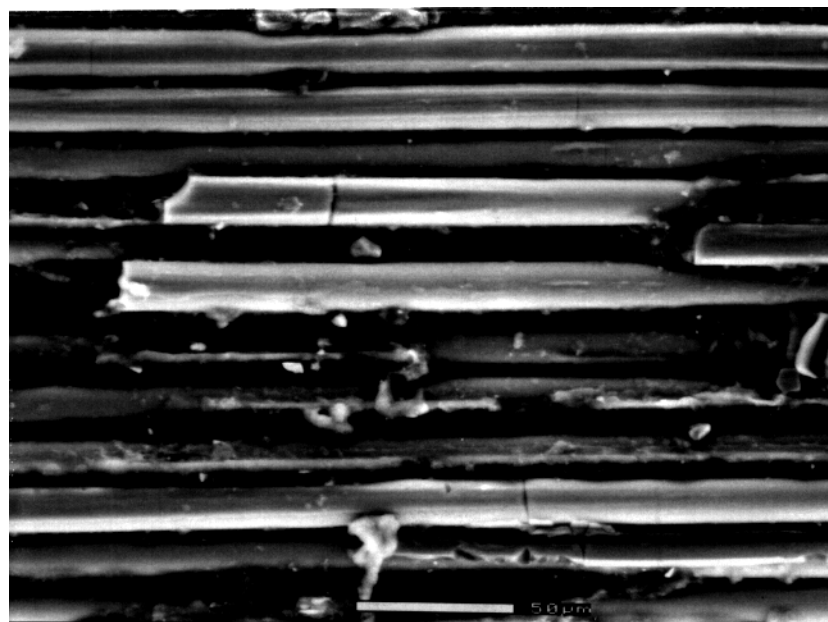


(b)

Figure 4.8 Photomicrographs of transverse tension fracture surface of glass-epoxy composite laminates with about 70% volume fraction of E-glass fibers. (a) Type A specimens; (b) Type B specimens; (c) Type C specimens; (d) Type D specimens. Bars represent 50 μm . (continued on following page)



(c)



(d)

Figure 4.8 Continued from previous page.

CHAPTER 5

DISCUSSION

It can be seen from the results given in Chapter 4 that only the contribution of energy dissipation from the interface or the interfacial damping can be related to interfacial adhesion strength. This interfacial damping factor was reported by Zorowski and Murayama [14], and is described in Chapter 2 (Equation 2.18); a modified rule of mixtures (ROM) equation was applied to obtain the damping factor for ideal (strong) interfacial bonding (Equation 2.18b). This equation has been used throughout the current research and thus it is necessary to obtain a better understanding of it.

5.1. Rule of Mixtures as Applied to the Damping Factor

Equation 2.18b can be derived using many different approaches. One of them involves in a simple and basic method described as follows.

It was noted in Section 2.2.1 that the vibration damping of a single component material can be viewed as a single element by considering the elastic contribution as a spring of constant k and the damping contribution as a dashpot of viscosity c . The free vibration of a single element can thus be considered as the spring-mass-dashpot system shown in Figure 5.1. From the free-body diagram (Figure 5.2), the equation of motion of the system, similar to Equation (2.3), can be written as

$$m\left(\frac{d^2x}{dt^2}\right) + c\left(\frac{dx}{dt}\right) + kx = 0 \quad (5.1)$$

Here x is the position coordinate that varies with the time t . Dividing throughout the equation by m and letting

$$\omega_0 = \sqrt{\frac{k}{m}} \quad \text{and} \quad \zeta = \frac{c}{2m\omega_0}$$

where ω_0 is the natural frequency, the equation of motion can then be written as

$$\left(\frac{d^2x}{dt^2}\right) + 2\zeta\omega_0\left(\frac{dx}{dt}\right) + \omega_0^2x = 0 \quad (5.2)$$

The solution to which is given as Equation (2.4).

This model can be extended to a two element spring-mass-dashpot system if two component materials are adhered with a perfect bond and vibrated at the same natural frequency. The system and the free-body diagram, in this case, are illustrated in Figures 5.3 and 5.4, respectively.

If it is assumed that the combined spring-mass-dashpot system obeys the rule of mixtures (ROM), the differential equation for vibration damping then becomes

$$(V_1 m_1 + V_2 m_2) \left(\frac{d^2 x}{dt^2} \right) + (V_1 c_1 + V_2 c_2) \left(\frac{dx}{dt} \right) + (V_1 k_1 + V_2 k_2) x = 0 \quad (5.3)$$

assuming that the volume fractions of the two component materials in the system are V_1 and V_2 , respectively. Equation (5.3) can also be written in the following form:

$$\left(\frac{d^2 x}{dt^2} \right) + 2\zeta_c \omega_0 \left(\frac{dx}{dt} \right) + \omega_0^2 x = 0 \quad (5.4)$$

where $\omega_0 = \sqrt{\frac{V_1 k_1 + V_2 k_2}{V_1 m_1 + V_2 m_2}} = \sqrt{\frac{k_1}{m_1}} = \sqrt{\frac{k_2}{m_2}}$ and $\zeta_c = \frac{V_1 c_1 + V_2 c_2}{2(V_1 m_1 + V_2 m_2) \omega_0}$

By reorganizing the expression of ζ_c , and knowing that $k_c = V_1 k_1 + V_2 k_2$, ζ_c can be rewritten as:

$$\begin{aligned} \zeta_c &= \frac{V_1 c_1}{2(V_1 m_1 + V_2 m_2) \omega_0} + \frac{V_2 c_2}{2(V_1 m_1 + V_2 m_2) \omega_0} \\ &= \frac{V_1 c_1}{2m_1 \omega_0} \frac{k_1}{\left(V_1 k_1 + V_2 \frac{m_2}{m_1} k_1 \right)} + \frac{V_2 c_2}{2m_2 \omega_0} \frac{k_2}{\left(V_1 \frac{m_1}{m_2} k_2 + V_2 k_2 \right)} \\ &= \frac{V_1 c_1}{2m_1 \omega_0} \frac{k_1}{(V_1 k_1 + V_2 k_2)} + \frac{V_2 c_2}{2m_2 \omega_0} \frac{k_2}{(V_1 k_1 + V_2 k_2)} \\ &= V_1 \frac{c_1}{2m_1 \omega_0} \frac{k_1}{k_c} + V_2 \frac{c_2}{2m_2 \omega_0} \frac{k_2}{k_c} \\ &= V_1 \zeta_1 \frac{k_1}{k_c} + V_2 \zeta_2 \frac{k_2}{k_c} \end{aligned}$$

Therefore,

$$\zeta_c = \frac{V_1 \zeta_1 k_1 + V_2 \zeta_2 k_2}{k_c} \quad (5.5)$$

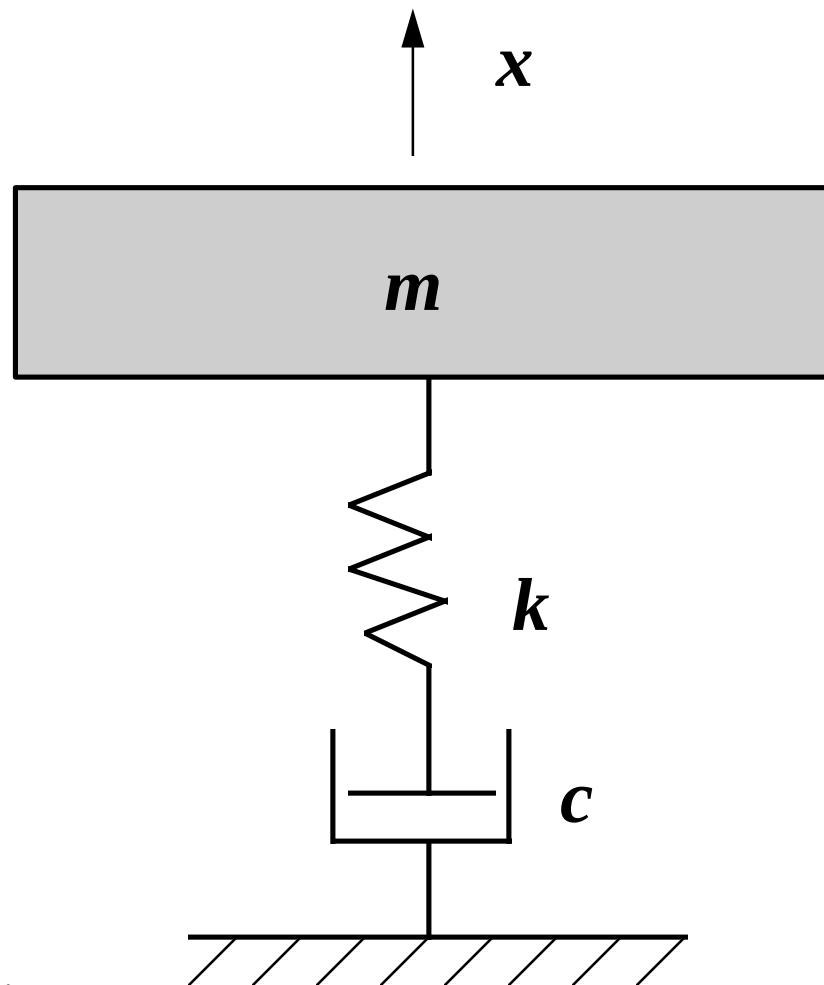


Figure 5.1 Single element of spring-mass-dashpot system.

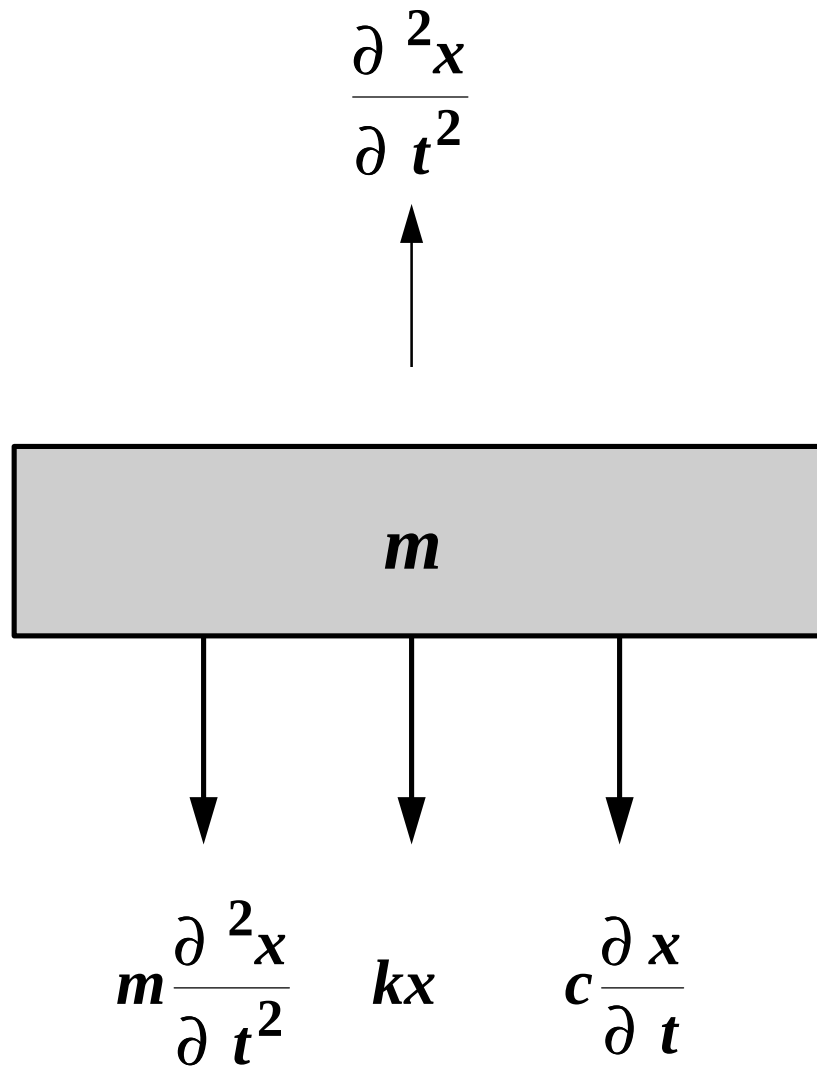


Figure 5.2 Free-body diagram of single element of spring-mass-dashpot system.

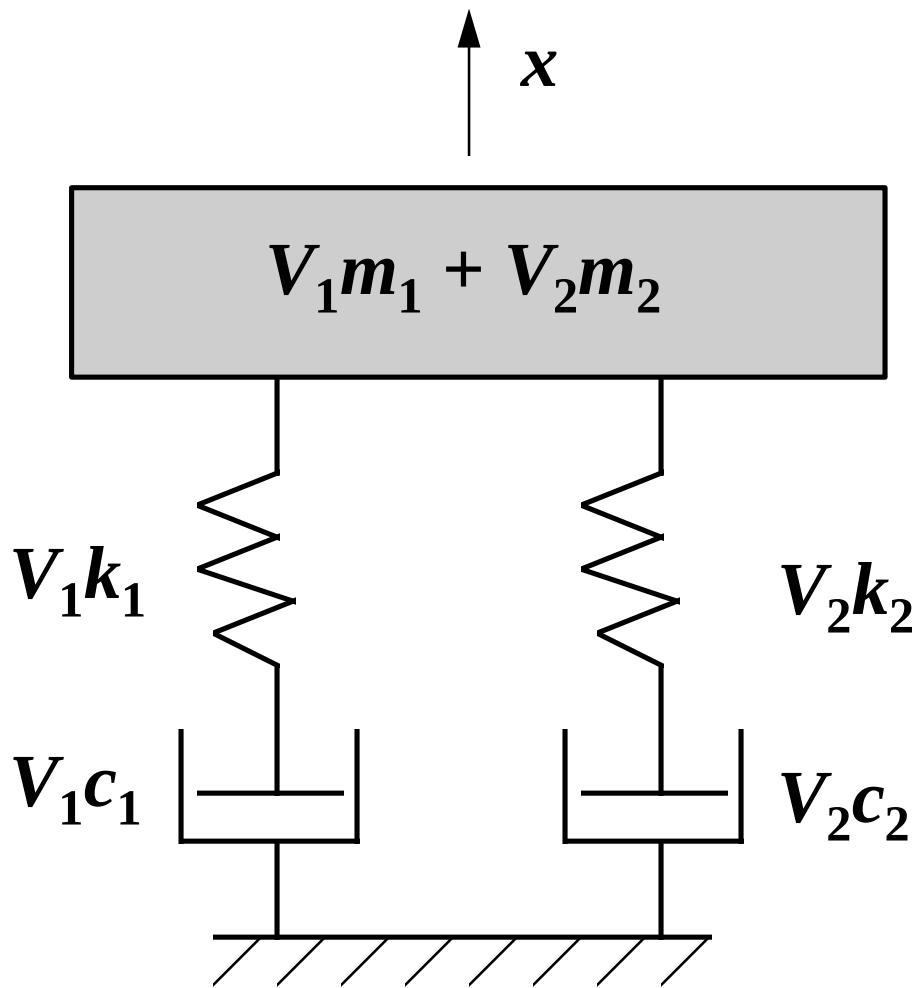


Figure 5.3 Two elements of spring-mass-dashpot system.

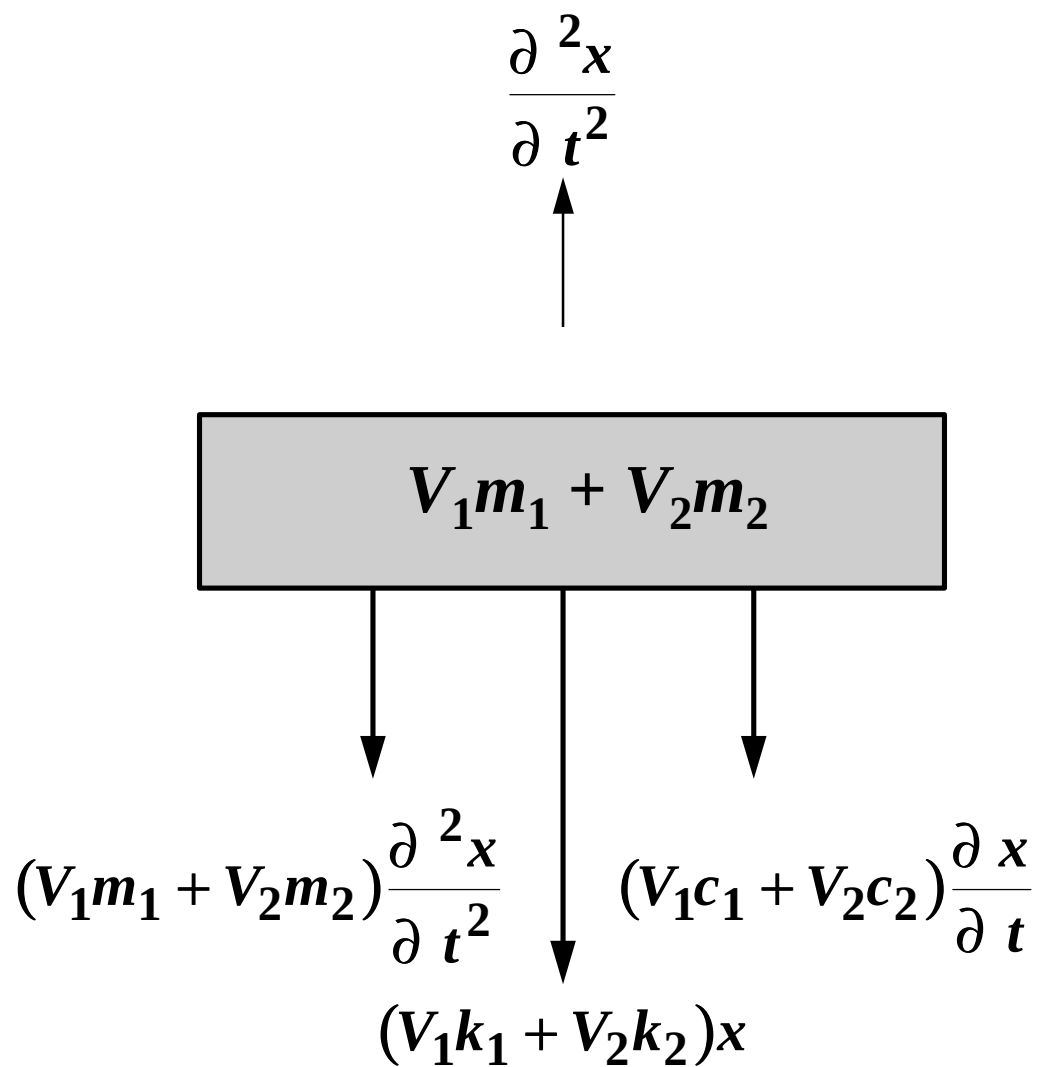


Figure 5.4 Free-body diagram of two elements of spring-mass-dashpot system.

It is known that the spring constant k and Young's modulus E are proportional to each other. For a cantilever beam, the correlation between k and E is [27]:

$$k = E \left(\frac{3I}{L^3} \right) \quad (5.6)$$

where I is the moment of inertia of the beam and L is the beam length. It has been suggested that damping factor ζ and $\tan \delta$ also be proportional to each other [25, 27], which can be seen from Equation (2.15). Thus, it is concluded that Equations (2.18b) and (5.5) are identical.

Equation (2.18b) can also be proven through definition of complex modulus, which is presented in Appendix A.

It has been emphasized that when fiber-matrix interfacial bond is not ideal, the interfacial energy dissipation should be taken into account, and the system will become more complicated, as schematically shown in Figure 5.5. It is not easy to completely solve this problem due to its complexity. However, it is reasonable to use the idea proposed by Zorowski and Murayama [14] or Equation (2.18) to view the interfacial damping as the total damping contributed from the composite material minus that contributed from each component material.

It is known that if the bond between the matrix and the reinforcement of a composite material is broken, the interface will dissipate energy when the composite is under vibration [49, 50]. The damping contributed from interfaces can be attributed to two mechanisms, i.e., interfacial slip or Coulomb friction which relates to interfacial energy dissipation from the already broken interface, and by interfacial debonding or cracking which corresponds to the newly created interfacial debonding during vibration. Zorowski and Murayama [14] analyzed the former mechanism and derived a proportional relationship between interfacial damping and the volume fraction of the components in a unidirectional fiber reinforced composite. However, it can be envisioned that the latter mechanism should dissipate more energy, as compared to the former. Therefore, if both mechanisms are involved in the interfacial damping, the latter mechanism will dominate. The following micromechanics model is thus derived according to this argument to see whether or not an inverse relationship between interfacial damping and interfacial adhesion strength holds, as experimentally found and stated in Chapter 4.

5.2. Micromechanics Model of Interfacial Damping

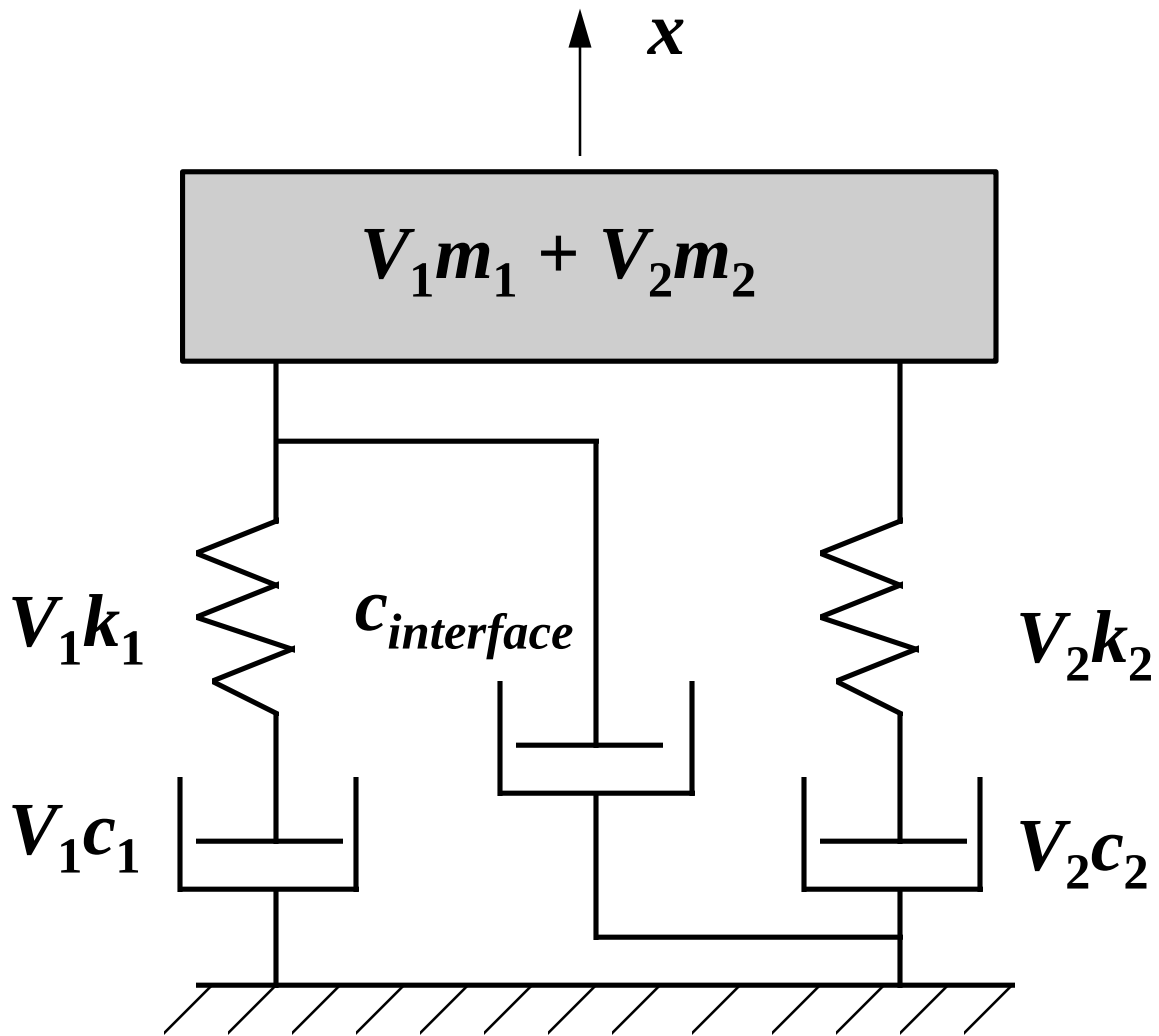


Figure 5.5 Two elements of spring-mass-dashpot system with a weak interface.

A schematic diagram of the model used to theoretically analyze the interfacial dissipative mechanism is depicted in Figure 5.6. A single fiber of constant cross section and properties is embedded in a matrix of lower modulus and of length equal to that of the fiber. Only loads parallel to the fiber axis are considered.

Based on the theory presented by Outwater Jr. [66], the analysis starts with the calculation of stress distribution in the fiber when it is axially loaded. For a partially debonded fiber, the mechanism that keeps the composite intact can be represented by the radial pressure surrounding and acting on the fiber, p . Any relative movement between the matrix and the fiber is resisted by the longitudinal component of this radial pressure. The free body diagram is shown in Figure 5.7. By balancing the forces acted on the segment, the following equation can be obtained:

$$\frac{dF_f}{dx} dx = \mu p \cdot 2\pi r_f dx \quad (5.7a)$$

where F_f is the force acting at the fiber, r_f is the fiber radius and μ is the friction coefficient between fiber and matrix.

Similarly, if the force F_f can cause interfacial debonding, then μp in Equation (5.7a) can be replaced by the interfacial shear strength of the composite, τ , which is that necessary to pull the fiber from the matrix. The equation then becomes

$$\frac{dF_f}{dx} dx = \tau \cdot 2\pi r_f dx \quad (5.7b)$$

Equations (5.7a) and (5.7b) represent Coulomb friction and interfacial debonding caused by F_f , respectively. Note that in both cases, F_f is assumed to be constant which is reasonable for small friction or debonding region. The total effect is the superposition of the two equations. However, as just mentioned in the previous section, when both mechanisms are involved in the interfacial damping, the latter mechanism will dominate because more energy is dissipated. Therefore, the energy dissipation part in Equation (5.7a) can be ignored and the energy consideration can be focused on the force balance in Equation (5.7b). By rearranging Equation (5.7b), we obtain:

$$\frac{dF_f}{dx} = 2\pi r_f \tau \quad (5.8)$$

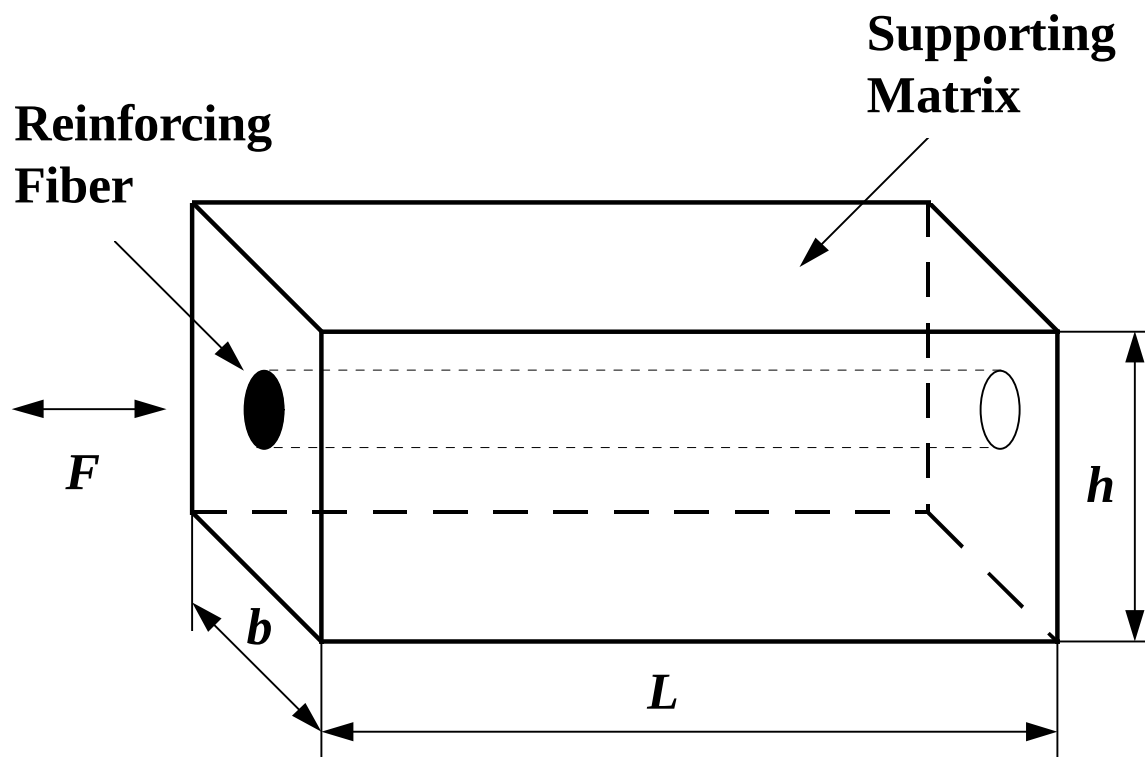


Figure 5.6 Simulated fiber reinforced composite model.

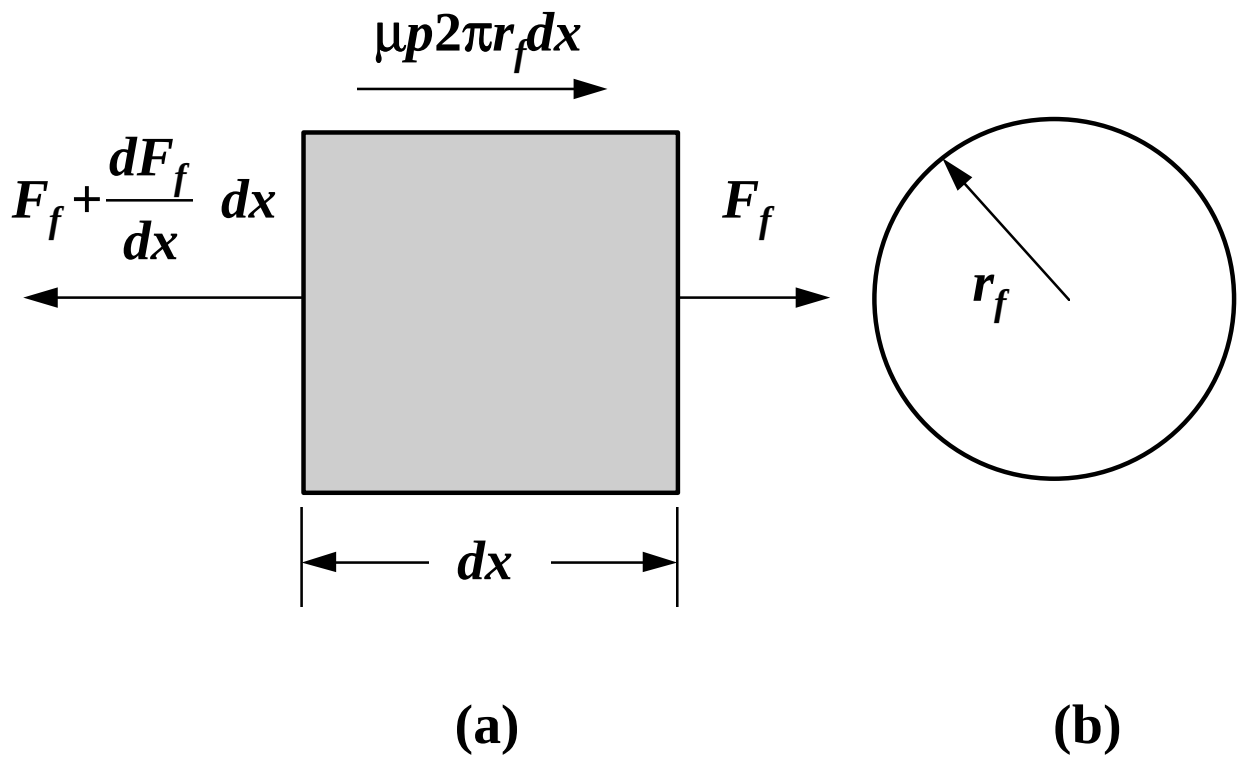


Figure 5.7 Forces acting on an element of a fiber. (a) Force balance in a fiber element; (b) Cross section of the fiber.

This equation can be easily solved to give solution

$$F_f = 2\pi r_f \tau x \quad (5.9)$$

Equation (5.9) is identical to Equation (2.1) in which debonding force was defined as F_d .

According to Equation (5.9), the debonding region can be estimated as:

$$x = \frac{F_f}{2\pi r_f \tau} \quad (5.10a)$$

The maximum debonding region is caused by the maximum load on fiber $F_f^{\max}$ when an alternating load is applied to the composite, which is true in the case of vibration,

$$x_0 = \frac{F_f^{\max}}{2\pi r_f \tau} \quad (5.10b)$$

For a cantilever beam, if there is interfacial debonding, it usually occurs at the free end of the beam because shear strain (and stress) is high at that location [67]. In this case, the force balance is shown in Figure 5.8.

Forces F_f and $F_f^{\max}$ can be found from bending load P of a cantilever beam as shown in Figure 5.9. When the beam is deflected by a load P , the axial (shear) component of the load can be obtained as

$$F = F_f = P \sin \theta_0 \approx P \theta_0 = \frac{P^2 L^2}{2E_c I_c} \quad (5.11)$$

where I_c is the moment of inertia of the composite beam. Therefore, the maximum load carried by fiber, $F_f^{\max}$, which corresponds to the maximum bending load, $P_{\max}$, can be expressed quantitatively as

$$F_f^{\max} = \frac{P_{\max}^2 L^2}{2E_c I_c} \quad (5.12)$$

Provided the energy dissipation in a cycle ΔW is small compared with the maximum stored elastic energy W of the system, which is true for most vibration damping situations, the loss coefficient η , or $\tan \delta_{in}$, may be estimated by Equation (2.15).

Assuming the stress in the fiber gradually approaches the maximum value (or P gradually reaches to $P_{\max}$) during a vibration cycle, interfacial debonding and slippage between fiber and

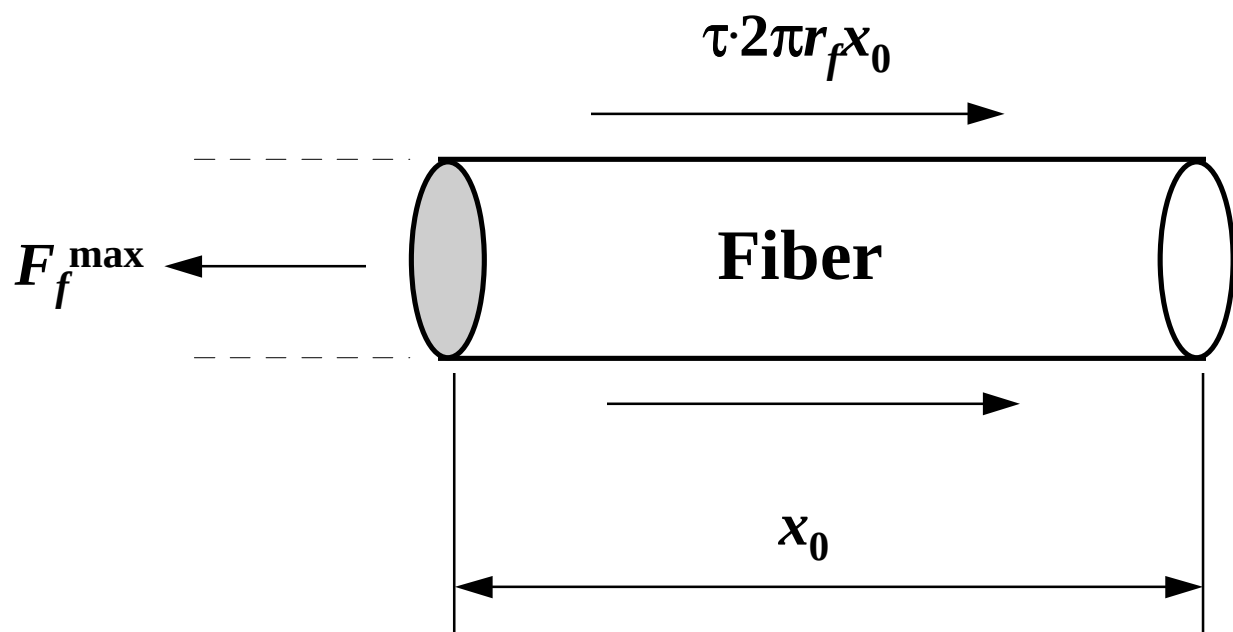


Figure 5.8 Free-body diagram of the free end of a cantilever beam.

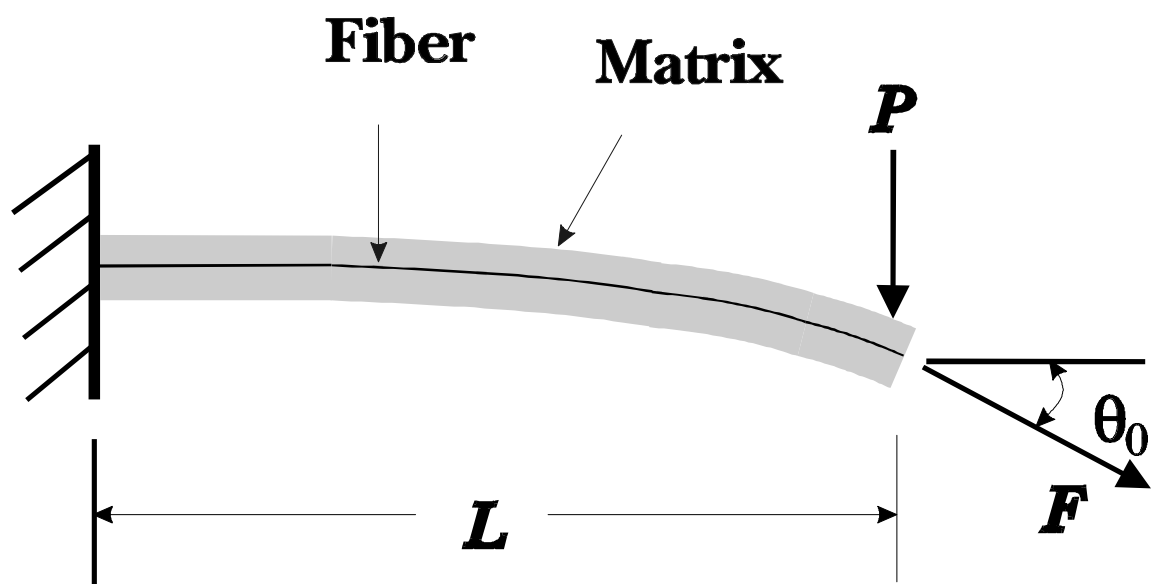


Figure 5.9 Force acting on a cantilever beam.

matrix will dissipate energy. Since the integration is performed over a complete cycle about the origin, the energy dissipated per cycle is given by

$$\Delta W = 2F_f^{\max} x_0 \quad (5.13)$$

Substituting Equations (5.10b) and (5.12) into Equation (5.13) yields

$$\Delta W = 2 \frac{P_{\max}^2 L^2}{2E_c I_c} \frac{\frac{P_{\max}^2 L^2}{2E_c I_c}}{2\pi r_f \tau} = \frac{P_{\max}^4 L^4}{4\pi r_f \tau (E_c I_c)^2} \quad (5.14)$$

The potential energy W for a sample subjected to strain ε can be calculated as follows

$$W = \int_0^{\varepsilon_{\max}} (\sigma_c A_c) \cdot d\varepsilon L = A_c L \int_0^{\varepsilon_{\max}} E_c \varepsilon d\varepsilon = \frac{1}{2} A_c L E_c \varepsilon_{\max}^2 \quad (5.15a)$$

where L is the sample length and σ_c , A_c and E_c are the stress, cross-sectional area and Young's modulus of the composite, respectively. For a cantilever beam, $\varepsilon_{\max} = \frac{P_{\max}^2 L^4}{15(E_c I_c)^2}$ [68], and we obtain

$$W = \frac{1}{2} A_c L E_c \left(\frac{P_{\max}^2 L^4}{15(E_c I_c)^2} \right)^2 \quad (5.15b)$$

By substituting Equations (5.14) and (5.15b) into Equation (2.15), and by knowing that $I_c = \frac{bh^3}{12}$ we obtain

$$\tan \delta_{in} = \frac{\Delta W}{2\pi W} = \frac{25E_c b h^5}{64\pi^2 \tau r_f L^5} \quad (5.16)$$

where b is the sample width and h is sample thickness.

If the number of fibers that can cause debonding in the composite is n , Equation (5.16) will be changed to

$$\tan \delta_{in} = n \frac{25E_c b h^5}{64\pi^2 \tau r_f L^5} \quad (5.17)$$

For the vibration of a cantilever beam, E_c can be calculated according to the vibration theory [29]

$$E_c = \frac{m_c \omega_c^2 L^4}{1.875^4 I_c} = \frac{12(\rho_f V_f + \rho_m V_m) \omega_c^2 L^4}{1.875^4 h^2} \quad (5.18)$$

where ω_c is the resonant frequency at the first mode of vibration, m_c is the mass of the composite per unit length, and ρ is the density.

Substituting Equation (5.18) into Equation (5.17) we obtain, after rearranging

$$\tau = B \frac{nbh^3(\rho_f V_f + \rho_m V_m) \omega_c^2}{\tan \delta_{in} r_f L} \quad (5.19)$$

where

$$B = \frac{75}{16 \times 1.875^4 \pi^2}$$

Equation (5.19) clearly shows the inverse relationship between $\tan \delta_{in}$ and τ , which is as expected.

There is another issue that should be clarified. It has been mentioned in Chapter 2 that when a cantilever beam is excited by a pre-displacement, the beam is vibrated with a frequency which is primarily composed of its first mode of vibration, and the response is the linear combination of all of the modes because it is not vibrated with an exact resonant frequency. In a related experiment, it has been found that the first mode of vibration is dominating for a cantilever beam vibration [69]. It is thus reasonable to use the first mode of vibration in the model derivation.

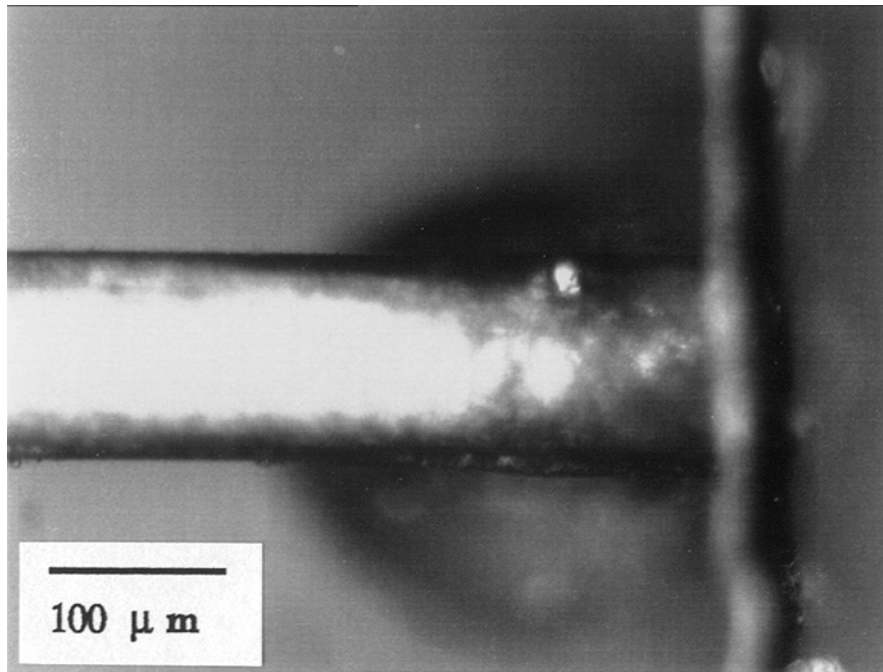
5.3. Observation of Cracks at the Free End of Composite Cantilever Beams

It is now appropriate to apply Equation (5.19) to the experimental results. However, before this occurs, it is necessary to verify the key assumption made in the derivation of the model, i.e., that interfacial debonding occurs during vibration. An empirical relationship between debonding length as a function of number of cycles was developed as a means to experimentally verify this assumption. According to the derived model, the debonded interfacial region of the composite after each run of vibration with a certain predisplacement is estimated to be approximate 1 to 2 μm . Therefore, the total debonded length after vibration for about 100 times should be around 0.1 to 0.2 mm assuming that the same debonding length is obtained for each

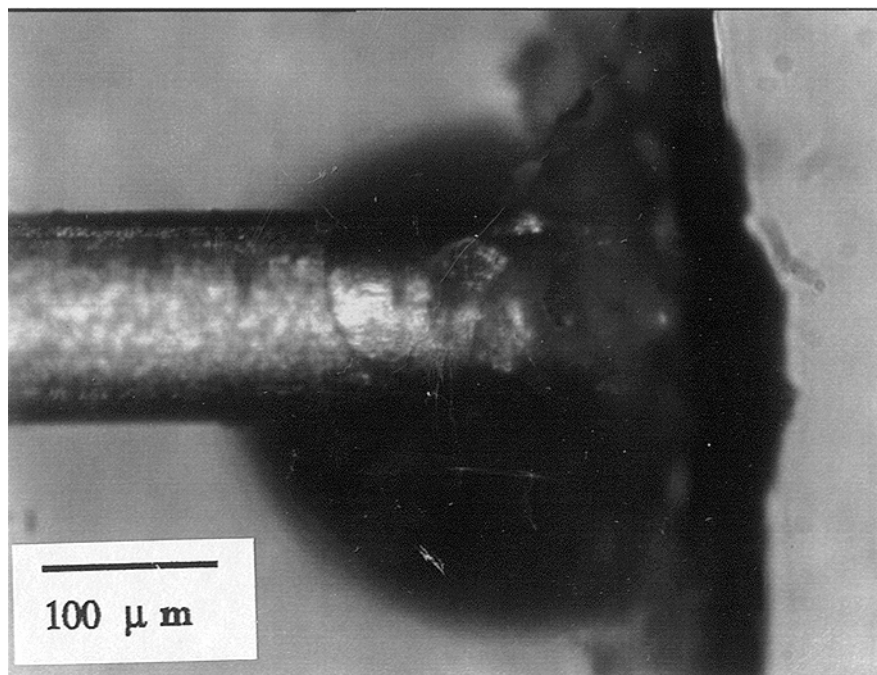
run. Several samples were used to examine this effect. Figure 5.10 clearly shows the debonding crack at the free end of a single molybdenum wire reinforced epoxy composite cantilever beam. The morphology of the crack is similar to that of debonded microbond samples which were also observed using the microscope for comparison. However, these observations are not conclusive. In fact, it has been found that it is difficult to observe cracks such as those shown in Figure 5.10 when simply doing vibration on single fiber (wire) reinforced composite samples. The cracks could be only observed in approximately 20% of the samples tested. Meanwhile, it brings out another question, i.e., whether the observed crack was initiated by bending the cantilever beam (it happened in the process of setting the predisplacement of the beam) or whether it was caused by the vibration process afterwards. Unfortunately, as just mentioned above, it is difficult to find a crack in single wire samples, and it is hard to distinguish the two process with a single specimen. To further verify the assumption that there is interfacial debonding during vibration, sandwich cantilever beam specimens consists of two pieces of polyethylene plate glued with double sided transparent tape were fabricated. Three sandwich specimens were prepared and pressed under a uniform weight for 36 hours. One of the specimens was then used as a control sample which was not tested any further. The second specimen was manually bent twenty times without vibration. The third, however, was mechanically vibrated twenty times after having been subjected to the same bending displacement as the second specimen. The free end of all cantilever beam specimens were observed under a microscope, and the micrographs of the three specimens are shown in Figure 5.11. The debonding cracks can be clearly viewed from the optical interference patterns. Note that it is difficult to find a crack in the control sample, and the specimen subjected to the vibrations exhibits the longest debonding crack. This is an indirect evidence that the model can indeed be applied to predict the interfacial adhesion strength.

5.4. Comparison of Calculated and Measured Interfacial Adhesion Strengths

After applying the model derived above, the interfacial adhesion strength can be calculated from interfacial damping value $\tan \delta_{in}$ according to Equation (5.19). For the single fiber (wire) reinforced epoxy composites used in the current study, the number of debonded fibers n is simply equal to 1. By utilizing the necessary data listed in Tables 4.5 and 4.7, predicted values of τ_{calc}

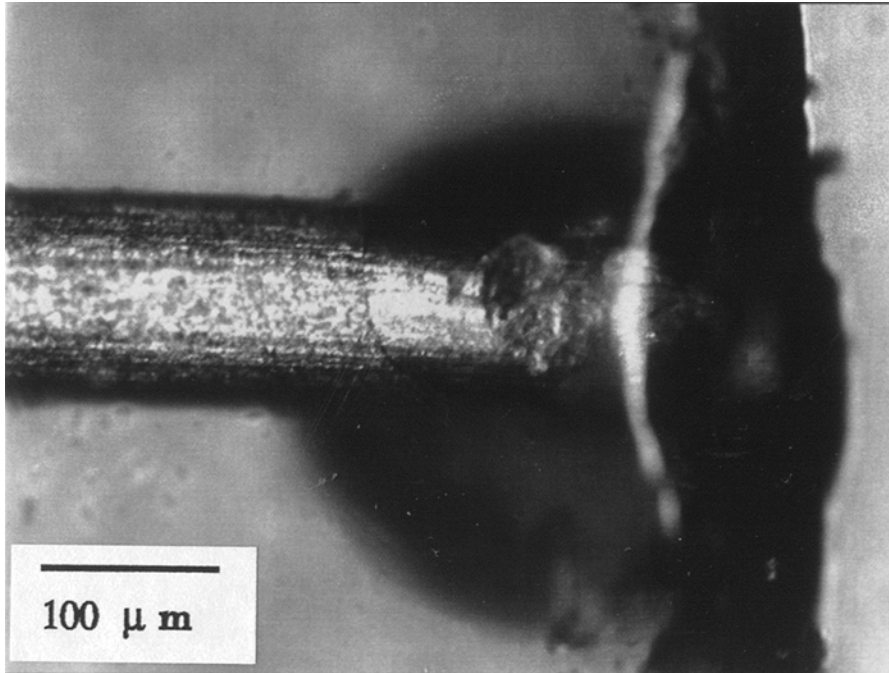


(a)



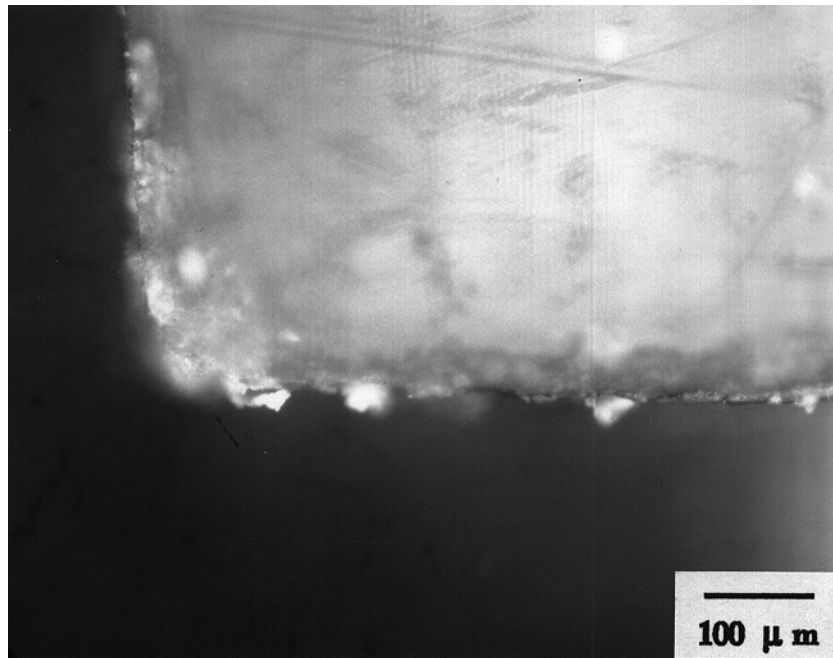
(b)

Figure 5.10 Micrographs of a free end of single molybdenum wire reinforced composite cantilever beam. (a) Before the vibration; (b) After vibration for 100 times; (c) After vibration for 200 times. (continued on following page)



(c)

Figure 5.10 Continued from previous page.

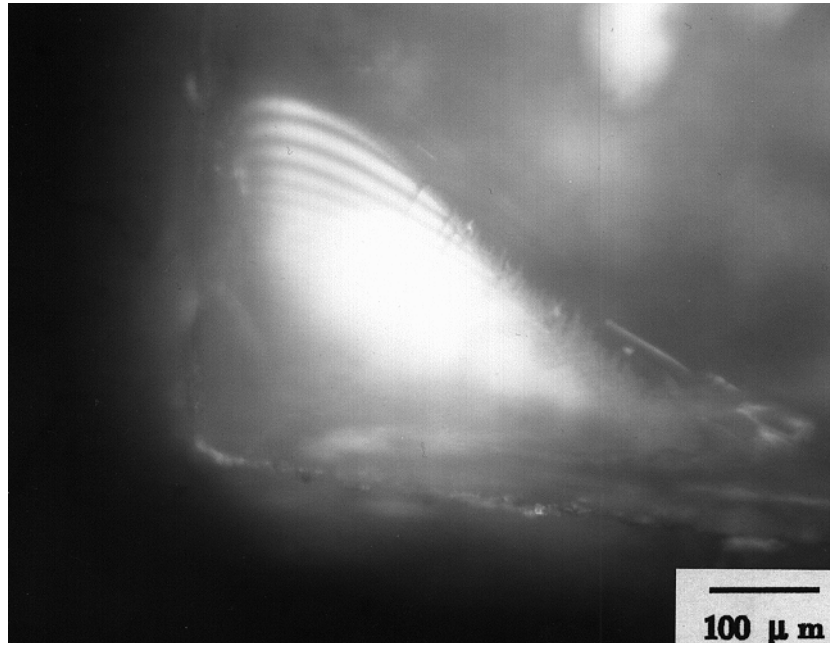


(a)



(b)

Figure 5.11 Micrographs of free ends of sandwich composite cantilever beams.
 (a) Without any deformation; (b) After bending for 20 times;
 (c) After vibration for 20 times. (continued on following page)



(c)

Figure 5.11 Continued from previous page.

were obtained, which are given in Table 5.1. By comparing the values of τ_{calc} in Table 5.1 and $\tau_{measure}$ in Table 4.1, it is found that reasonable agreement is shown between prediction and experiment. This comparison is graphically illustrated in Figures 5.12 through 5.14.

It is interesting to note that different fibers (wires) have various adhesion properties with Epon 828TM epoxy resin used in the current study. It is understandable that the wires coated with silicone mold release exhibit lower interfacial adhesion strength because of the weakening of the wire-matrix interface. The reason for stronger interfacial adhesion in metallic wire-reinforced composites than that in glass fiber-reinforced composite can be attributed to the different surface roughness the fiber or wires. The glass fiber has a smoother surface than metallic wires resulting in poorer interlocking between fiber and epoxy resin, and this could be the main reason for lower interfacial adhesion strength.

Table 5.2 lists the reported interfacial adhesion strengths for glass fiber-reinforced epoxy composite obtained from microbond single fiber pull-out tests. It can be seen that the reported data differ from one research group to another. It indicates that direct comparison of experimental data from different sources is not advisable. It is clear that the interfacial adhesion strength obtained from the current study (Table 4.1) is significantly lower than those in Table 5.2. This may be due to the larger glass fiber diameter used in the present research (about 10 times larger than usual), which is mathematically predicted by Equation (5.19). Different fiber surface conditions may also affect the results.

It should also be pointed out that the interfacial damping data of the single fiber (wire) reinforced epoxy composites presented above were obtained from only 75% of specimens actually tested. Approximately 25% of the tested samples provided some unreasonable negative interfacial damping values. This phenomenon may not be surprising because of the following reasons.

First, debonding is not guaranteed to occur at the fiber-matrix interface for a single fiber (wire) composite. When interfacial damping data are close to zero or even negative, it likely reflects that an ideal interfacial bond exists in those specimens during the vibration. In other words, no interfacial debonding occurred when vibration of those specimens was carried out.

Second, it has been widely reported [16-17, 72-74] that the bonding between fiber and matrix in a composite usually is not simply contacted by a cohesive force developed on the interface of the two main phases. Indeed it has been shown that the interrelationships between

Table 5.1 Damping factors contributed from interface and calculated interfacial adhesion strength of single fiber (wire) reinforced composites

Fibers	Fiber Diameter (μm)	$\tan \delta_{in}$ (10^{-3})	τ_{calc} (KPa)
Mo	127	0.58 (± 0.27)**	1145 (± 480)
Mo*	127	0.79 (± 0.30)	793 (± 458)
Cu	100	0.58 (± 0.18)	1471 (± 736)
Cu*	100	1.29 (± 0.51)	706 (± 231)
W	100	0.34 (± 0.13)	1328 (± 495)
W*	100	1.67 (± 0.76)	482 (± 224)
Glass	120	0.80 (± 0.21)	419 (± 161)

* With silicone mold release.

**Numbers in parenthesis represent ± 1 standard deviation.

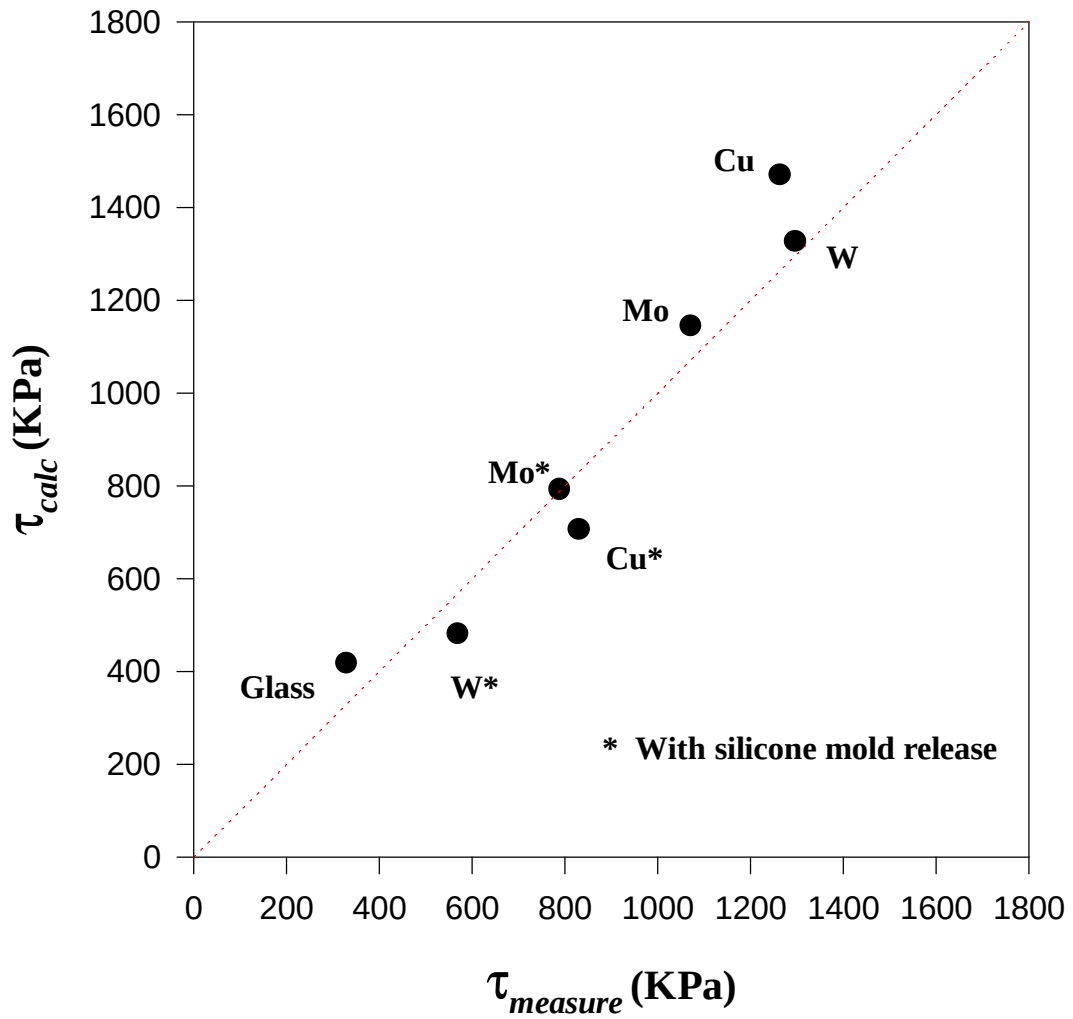


Figure 5.12 Comparison of average values of τ_{calc} and $\tau_{measure}$ of single fiber (wire) reinforced composite

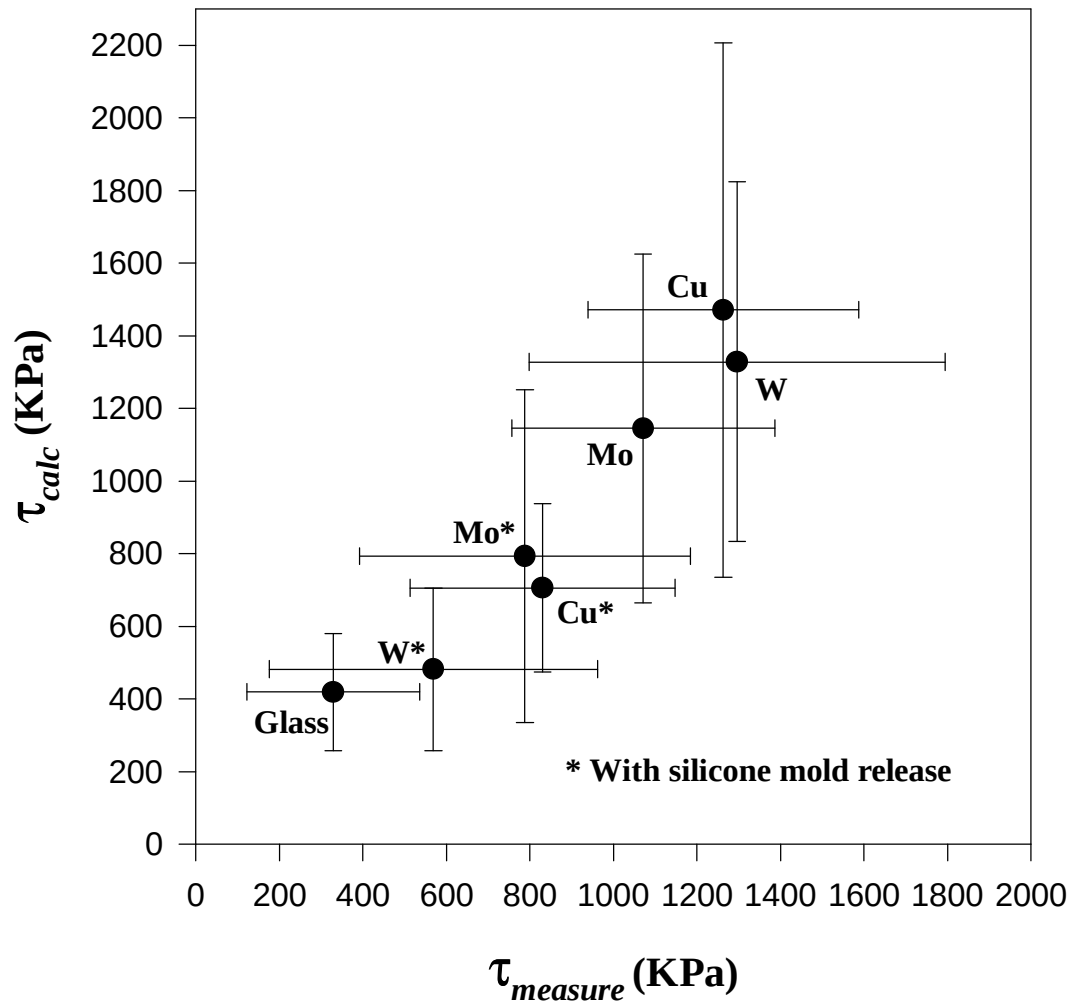
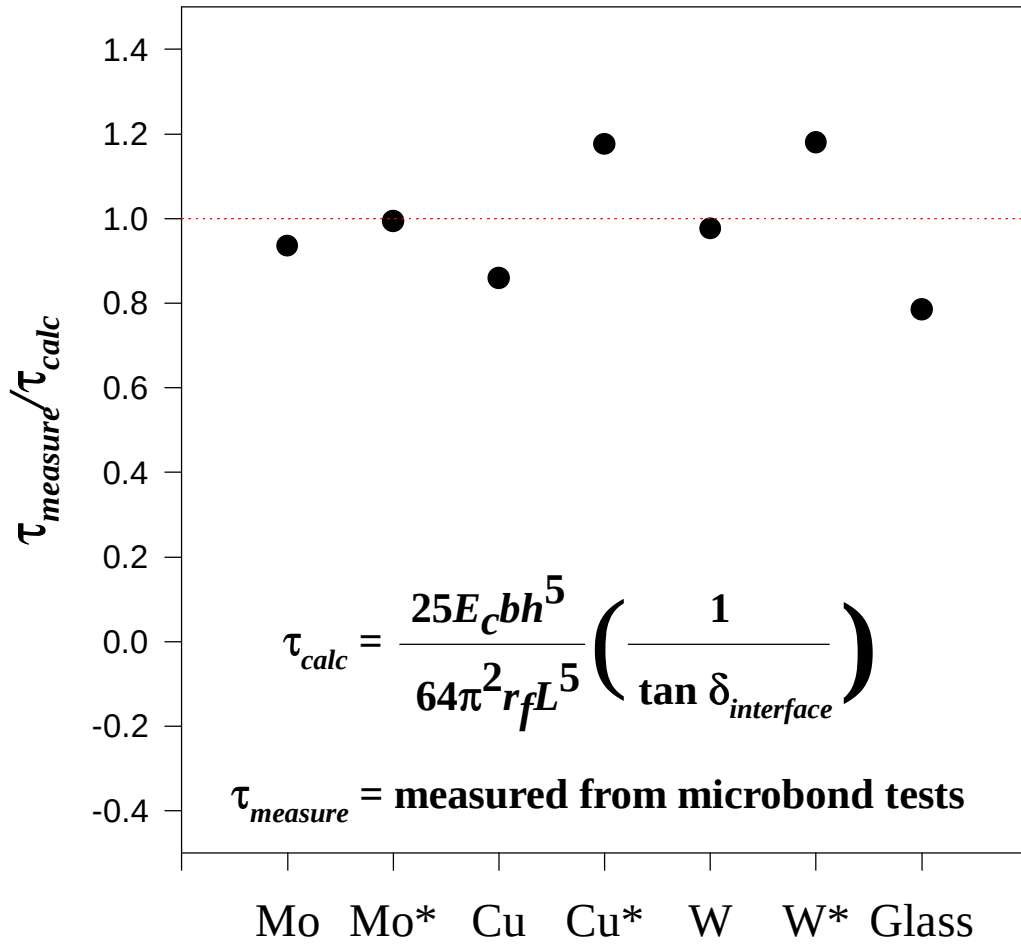


Figure 5.13 The same curve as shown in Figure 5.12. Error bars represent ± 1 standard deviation.



* With silicone mold release

Figure 5.14 Comparison of average values of τ_{calc} and $\tau_{measure}$ of single fiber (wire) reinforced composites with another method to analyze the data.

**Table 5.2 Reported interfacial adhesion strengths of single glass fiber-reinforced epoxy
composite obtained by pull-out tests**

τ (MPa)	Reference
11-33	[4]
19	[70]
3.5-4.5	[71]

fiber, interface and matrix create a complex region termed an interphase and it is obviously inhomogeneous. According to the illustration made by Drzal [74], this interphase includes not only the region of contact between fiber and matrix (the interface) but also a region on both sides of the interface. The local properties start to change from the bulk properties in the direction of the interface. In this region, chemical and morphological features are different from the component materials. Meanwhile, impurities, unreacted polymer components, non-polymerized matrix additives, voids, and adsorbed gasses can also be found in the interphase. The resultant interphase thus can be a very complex material which can not easily be analyzed by a single parameter models. Therefore, more complicated micromechanics model may need to be taken into account, which will include the interphase to form a three phase model. This will be more realistic for real composites in which sizing and silane are usually applied to modify the interfacial adhesion and may form many interphase regions.

For E-glass fiber reinforced epoxy composite laminates, it is difficult to detect the number of debonded fibers, n , after failure. Equation (5.19), therefore, can not be directly applied to predict the interfacial adhesion strength at the present time. However, the inverse relationship between energy loss from the interface, $\tan \delta_{in}$, and interfacial adhesion strength, τ should still hold. This relationship should be true even if there is no instant debonding occurring during the vibration because more energy dissipation from the interface will reflect weak interfacial slippage.

Equation (2.18) shows that a higher value of $\tan \delta_{in}$ reflects poor adhesion in the system. Results shown in Figures 4.5 and 4.6 indicate that Type A specimens appear to have the weakest fiber-matrix interfacial adhesion among the four composite systems. However, Type C specimens, which contains 366 size with silane, show the strongest interfacial adhesion. It is also interesting to note that the Type B specimens (having 366 size without silane) and Type D specimens (having 158 size with silane) seem to have an equal magnitude of interfacial adhesion.

In order to support the arguments, a comparison of the microstructures of different types of bonding mechanisms of the composites was carefully made for all the fracture surfaces (refer to the photomicrographs in Figure 4.7). It is obvious that 366 size with silane and 158B size with silane specimens show better fiber-matrix wetting than other types of specimens. Clean fiber-matrix interface is clearly observed in the untreated fiber specimens.

However, from Figure 4.4 it is obvious that values of σ_{tr} are almost identical for all four composite systems with about 30% volume fraction of E-glass fibers despite the $\tan \delta_{in}$ values showing the same trend as 50% and 70% volume fraction of E-glass fiber composite laminates. This can be explained as follows. If a composite specimen has 30% volume fraction of E-glass fibers, there is large amount of epoxy resin in the composite. When transverse tensile strength tests were conducted, the failure usually occurred in the matrix material, not at the fiber-matrix interface. In this case, therefore, the measured values of σ_{tr} represent indeed the tensile yield strength of the matrix material or the epoxy resin. It is thus even more interesting to note that the vibration damping method is more sensitive to the fiber-matrix interface, and is likely to be a more promising technique to detect the fiber-matrix interfacial adhesion.

It is known that composites with poor interfacial bonding exhibit a low interfacial adhesion strength, τ . It can easily be seen from results stated above that the observations from $\tan \delta_{in}$ are consistent with the results from τ , and both show the same magnitude of the interfacial bonding. In other words, $\tan \delta_{in}$ and τ show a strong inverse correlation. Some experimental data have been presented and published in the conference and technical journal [75, 76]. It has been shown that this relation is still valid when the interface is affected by moisture (refer to Appendix B).

CHAPTER 6

SUMMARY

Adhesion at the fiber-matrix interface in fiber-reinforced composites plays an important role in controlling the mechanical properties and overall performance of composites. Among the many available tests applicable to composite interfaces, the vibration damping technique has the advantage of being nondestructive as well as highly sensitive. A simple optical system was constructed for measuring the damping factor of uniaxial fiber-reinforced-polymer (FRP) composites in the shape of cantilever beams. Adhesion at fiber-matrix interfaces in single fiber (wire) reinforced epoxy-resin composites and E-glass-fiber reinforced epoxy-resin composite laminates was characterized by using the system.

The single fibers (wires) used in the current study included glass, copper, molybdenum and tungsten. The interfacial adhesion strengths measured from microbond pull-out tests are compared with measured interfacial damping factor of a single fiber (wire) composite cantilever beam. An inverse relationship between the damping characteristics of the fiber-matrix interface and interfacial adhesion strength of composites was found from the experimental results, which is consistent with a derived micromechanics model. It is noticed that the interfacial adhesion strength is dependent on the roughness of the fiber surface. A smooth fiber surface in the composite may lead to a low interfacial adhesion strength.

The study was also extended to multi-fiber composite laminates. Four different composite systems were selected. For each system, glass fibers with three different surface treatments were used at three different volume fractions. The interfacial damping factors of the composites were obtained by experiments from the optical system. It is noticed that the interfacial damping factor is proportional to the volume fraction of fibers. As the volume fraction of fibers in the composite increases, more fiber-matrix interfacial area is created, and more energy can be dissipated by the fiber-matrix interface. The obtained interfacial damping factors were correlated with transverse tensile strength, which is also a qualitative measurement of adhesion at the fiber-matrix interface. It is shown that the composite system which has the 366 size with silane exhibits the best fiber-matrix interfacial adhesion. The interfacial adhesion of the system which has the 366 size without silane turns out to be compatible with that of the system which has the 158B size with silane.

Samples with untreated fibers show the weakest interfacial adhesion among all samples. The experimental results showed that an inverse relationship between damping characteristics of the fiber-matrix interface and transverse tensile strength of composite laminates is also valid.

In conclusion, it has been shown that an inverse relationship exists between interfacial damping factor and interfacial adhesion strength for uniaxial fiber-reinforced polymer composites. The interfacial damping factor, therefore, can be used as a parameter to characterize the fiber-matrix adhesion of the composites.

The future work is suggested to complete the micromechanics model by considering more complicated situation, such as interphase and combination of interfacial slippage and crack growth during the vibration. More environmental experiments are also suggested.

APPENDIX A

DERIVATION OF EQUATION (2.18b) FROM DEFINITION OF COMPLEX MODULUS

Assuming that the complex modulus of a composite obeys rule of mixtures (ROM). i.e.,

$$E_c^* = E_f^* V_f + E_m^* V_m \quad (\text{A.1})$$

According to definition, Equation (A.1) can be rewritten as:

$$E_c' + iE_c'' = (E_f' + iE_f'')V_f + (E_m' + iE_m'')V_m \quad (\text{A.2})$$

By separating the real part and imaginary part from Equation (A.2), the following expression can be reached.

$$E_c' = E_f' V_f + E_m' V_m \quad (\text{A.3a})$$

$$E_c'' = E_f'' V_f + E_m'' V_m \quad (\text{A.3b})$$

According to the definition of $\tan \delta$, the following derivation should hold.

$$\begin{aligned} \tan \delta_s &= \frac{E_c''}{E_c'} = \frac{E_f''}{E_c'} V_f + \frac{E_m''}{E_c'} V_m \\ &= \frac{E_f'}{E_c'} \frac{E_f''}{E_f'} V_f + \frac{E_m'}{E_c'} \frac{E_m''}{E_m'} V_m \\ &= \frac{E_f'}{E_c'} \tan \delta_f V_f + \frac{E_m'}{E_c'} \tan \delta_m V_m \end{aligned}$$

Therefore,

$$\tan \delta_s = \frac{E_f'}{E_c'} \tan \delta_f V_f + \frac{E_m'}{E_c'} \tan \delta_m V_m \quad (\text{2.18b})$$

APPENDIX B

MOISTURE EFFECTS ON INTERFACIAL ADHESION IN COMPOSITES

As mentioned throughout this study, adhesion between the fiber and matrix in fiber-reinforced polymer composites plays an important role in controlling the mechanical properties and overall composite performance. It is even more crucial when such materials are exposed in an aggressive aqueous environment involving possibly alkalis, acids, and elevated temperatures. One of the issues that must be dealt with is the tendency of these material systems to absorb moisture while in service. Moisture absorption can have a deleterious effect on fiber-matrix interfacial adhesion and consequently on the overall behavior of the composites. A series of research work on this issue has been conducted, and was summarized by Springer [77]. Most of the reported work was aimed at the effect of moisture absorption on the whole composite [77, 78]. Only a few studies have been found that investigate the water effect on the composite interface with fragmentation tests [79]. To take advantage of the vibration damping technique and the simple optical system described in this study, the effect of moisture absorption on interfacial adhesion of glass-fiber reinforced epoxy-resin composites can also be investigated by relating the interfacial adhesion strength to energy dissipation contributed from the fiber-matrix interface.

This work has been in progress but not yet completed. The study focused on the periodic measurements of the damping of cantilever composite beam samples that were previously immersed in hot distilled water at 80°C. The measurements were made with the optical system described in Chapter 4. Four different composite systems containing three different glass-fiber surface treatments with about 30% volume fraction of fibers, which are the identical specimens used for fiber volume fraction effects, were tested. The pure epoxy specimens were also immersed in the hot distilled water and tested each time with the composite specimens together to obtain the instant damping factor of the matrix material. It is assumed that the damping characteristics of glass fibers do not change with exposure in the moisture.

The weight changes of the materials are plotted in Figure B.1. The weight change is defined as:

$$M(\%) = \frac{\text{Weight of specimen} - \text{Weight of dry specimen}}{\text{Weight of dry specimen}} \times 100 \quad (\text{B.1})$$

The results presented in Figure B.1 show the weight gains of the four types of composites at immersion times ranging from 0 to 1700 hours. It can be seen that at every test condition the weights of the specimens increased at first and leveled off for some length of time. These results indicate that, in addition to immersed time, both the initial rate of weight increase and the value at which the weights leveled off also depends on the material.

The interfacial damping factor, $\tan \delta_{in}$, was calculated for each test according to Equation (2.18). The results are plotted in Figure B.2. Generally, the results show increasing energy dissipation with water immersion time. Also, we observed a correlation between interfacial damping factor and interfacial adhesion strength — lower damping factors for higher adhesion strengths. For example, Type A composite had the highest interfacial damping factor and is expected to have the lowest interfacial adhesion strength because of the untreated fibers used in the material system. On the other hand, Type C composite had the lowest interfacial damping and was expected to have the highest interfacial adhesion strength because the fibers were treated with the 366 size and silane to improve the adhesion. Type C and D composites exhibited interfacial damping factors which lie in between those of A and B, and were expected to have intermediate interfacial adhesion strengths. Based on these results, it can be concluded that composites that contain the silane coupling agent are likely to retain their performance in hot-wet environments. However, it is noticed that a peak appears in Figure B.2 for all types of specimens utilized in the current study. By carefully analyzing the data, it can be found that the damping curves of the pure epoxy have an abnormal behavior in the peak region (immersion time from 24 to 300 hours). A typical damping curve of pure epoxy with 24 hours exposure is shown in Figure B.3.

The normal damping curve exhibits an exponential decay as shown in Figures 4.1 and 4.2. It is easy to directly obtain the damping factor or loss tangent ($\tan \delta$) from Equation (3.1) or by curve fitting using Equation (3.2). However, in the case of the abnormal curve illustrated in Figure B.3 which presents several “loops” with “peaks” and “valleys”, it is questionable to select which data to calculate $\tan \delta$ although the overall exponential decay can still be recognized in the curve. In the current study, the “peak” data in each “loop” were chosen to fit the decay curve. The obtained $\tan \delta$ thus is the minimum value comparing to any other selection. This choice

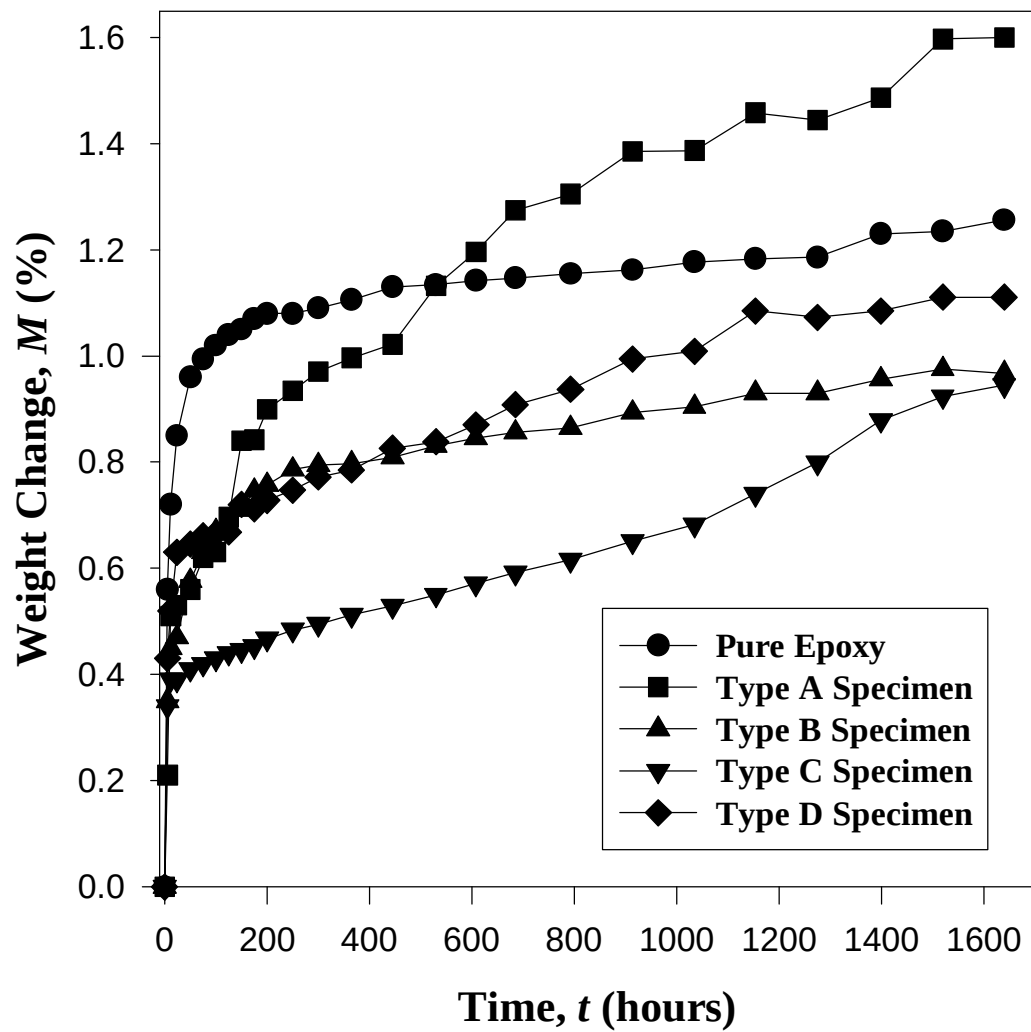


Figure B.1 Weight changes as a function of time of specimens immersed in distilled water at 80°C.

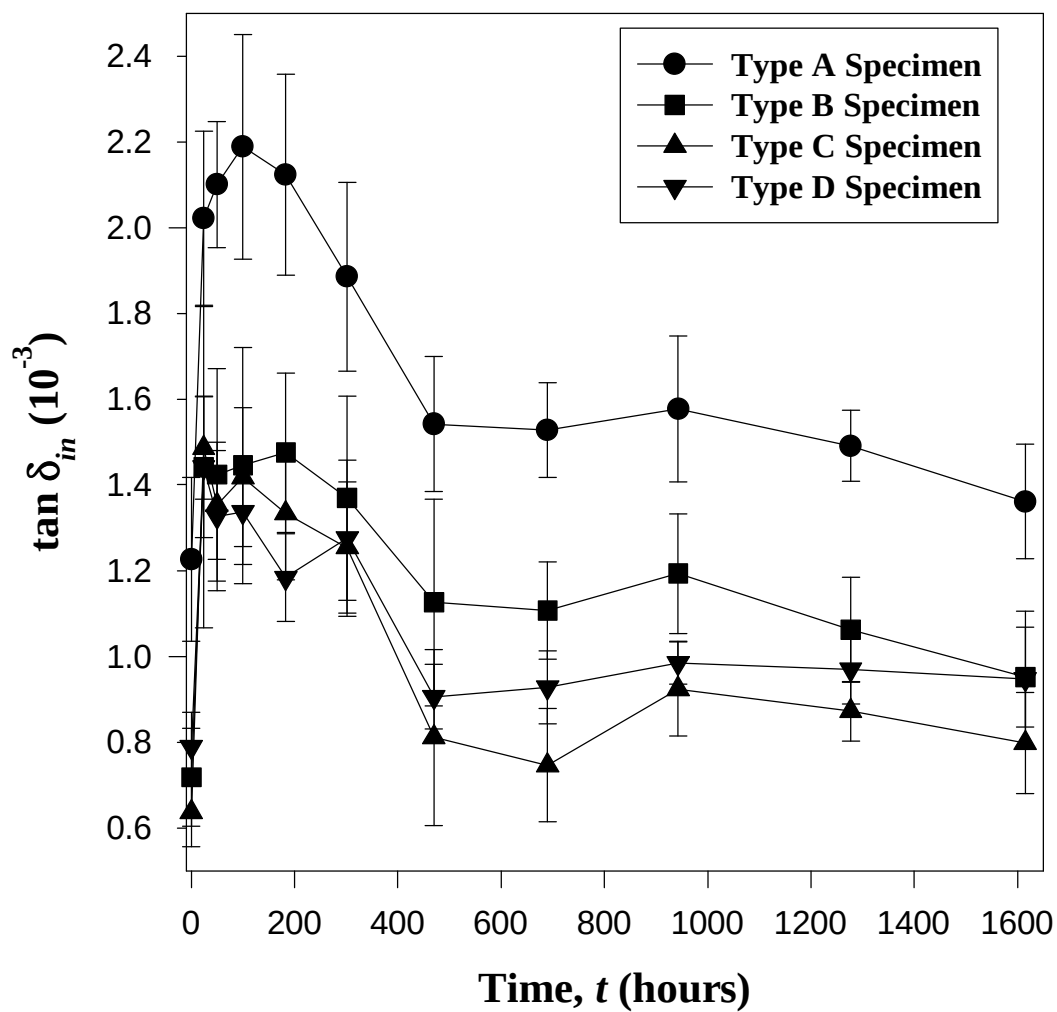


Figure B.2 Changes of interfacial damping as a function of time of specimens immersed in distilled water at 80°C.

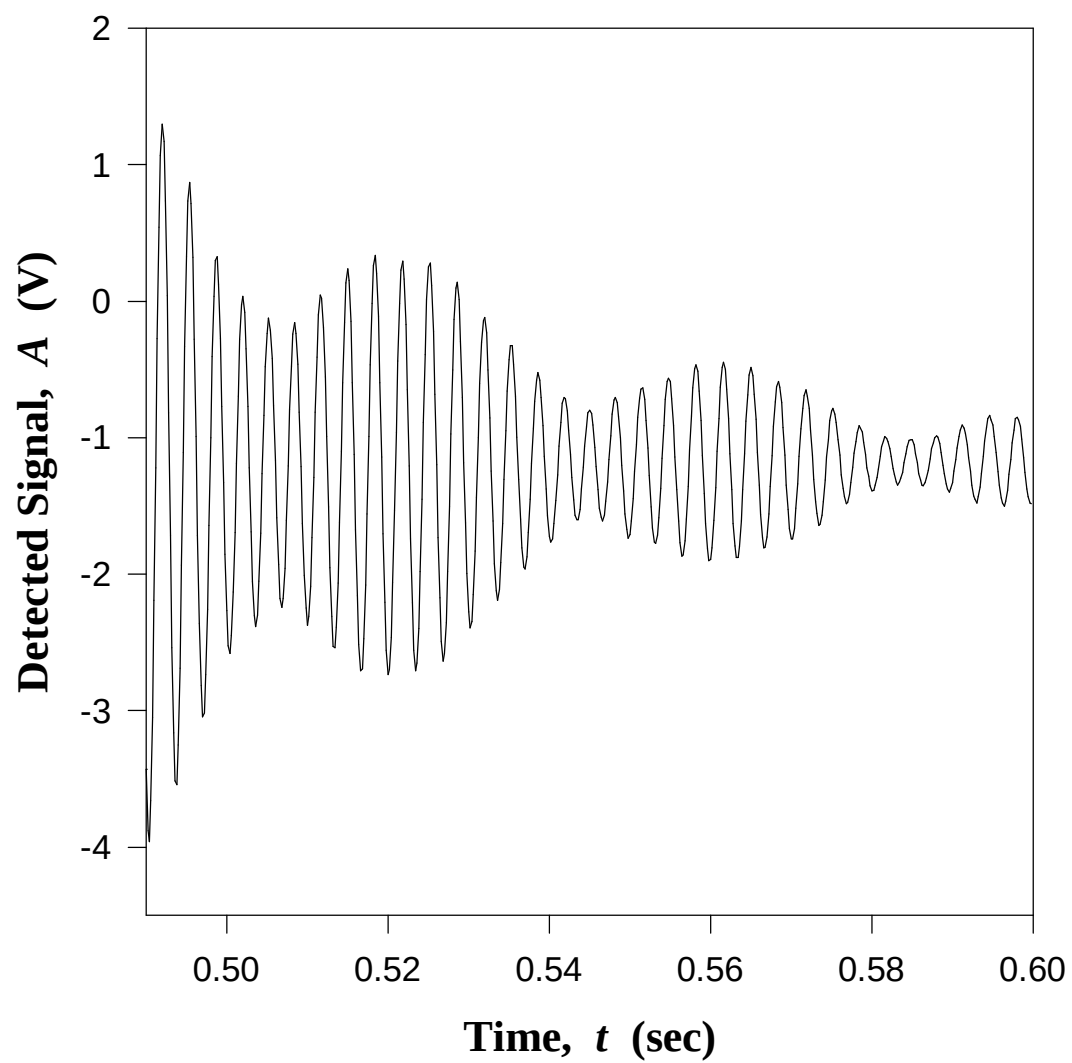


Figure B.3 A typical damping curve of pure epoxy immersed in distilled water at 80°C for 24 hours.

may not be physically sound. In fact, if the average data in the “loop” or the “valley” data were selected, the resultant $\tan \delta$ would be larger. When these larger values are applied, the interfacial damping factor $\tan \delta_{in}$ will become small according to Equation (2.18). Therefore, further research is suggested to be carried out for understanding the mechanism of the abnormal curve. The vibration damping technique will be successfully applied for characterizing the interfacial adhesion of composites with moisture effect as long as the water effect on the dynamic properties of pure epoxy is fully understood.

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