Study on the Relaxation Behavior of Bamboo Fiber as a Function of Temperature and Moisture Content

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The tensile relaxation behavior of bamboo fiber was evaluated. Bamboo fibers were extracted *via* a mechanical method and treated in alkaline solutions of sodium hydroxide. The relaxation tests of bamboo fibers were conducted by applying a constant strain at one end of a single bamboo fiber while the other end was fixed. The reaction force in the bamboo fiber was measured at the same time. The relaxation tests of the bamboo fibers were conducted under different temperature and moisture contents. The results showed that in the wet and/or high temperature conditions, the relaxation rate was faster than that in the dry and/or room temperature conditions. For a low strain, the bamboo fiber can be totally relaxed within 10 min under a high temperature. A nonlinear viscoelastic constitutive model was proposed to fit the relaxation curves, which could model the stress relaxation behavior of a bamboo fiber under different temperature or moisture content conditions. Compared with the experimental data, the model gave reasonable values for the relaxation behavior of the bamboo fiber.

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INTRODUCTION

Materials that exhibit both viscous and elastic characteristics are called viscoelastic materials. Amorphous polymers (plastic), fiber materials (silk, rayon), biological materials (skin), cement, concrete, ceramics, and glass all have viscoelastic behavior. The effects of material viscoelasticity on engineering applications cannot be ignored in some cases, especially for the architecture industry. For instance, the creep properties of cement and concrete must be taken into consideration in the procedures of design and analysis (Lakes and Lakes 2009); otherwise, excessive deformation will be generated and cause the structure to rupture.

Natural fiber composites contain natural fibers such as sisal, flax, jute, kenaf, wheat, rice, and bamboo used as the reinforcement (Young and Tsao 2015; Sukmawan *et al*. 2016; Jordan and Chester 2017; Chou and Young 2018; Hao *et al*. 2018; Sood *et al*. 2018; Chiu and Young 2020a,b). These natural fibers may have more obvious viscoelastic behavior than inorganic fibers (Kumar 2020; Kumar and Raja 2021; Kumar *et al*. 2021; Muthalagu *et al*. 2021; Raja and Kumar 2021; Shih and Young 2022). It is important to study the viscoelastic behavior of natural fibers being used as the reinforcement of composites.

One of the major behaviors of viscoelastic materials is relaxation. As a constant strain is applied to the material, the corresponding stress will decrease with time, which is called relaxation. The temperature and moisture content of natural fibers have influence on the relaxation behavior. Nakajima *et al*. (2011) found that as the temperature increased, the flexural modulus of a bamboo fiber decreased. The thermal softening of the structure is caused by the micro-Brownian motion of lignin. Iida *et al*. (2002) indicated that the stress relaxation ranges of wood at high temperature were larger than those at low temperature. The detailed relaxation trend was related to the heating rate and preheating. The relaxation range of the preheated sample was larger than that without preheating, and the relaxation rate was correlated to the heating rate. Wang *et al*. (2017) studied the rice and reported that the elastic modulus decreased as the moisture content increased, which meant that the relaxation rate was faster when the moisture content was higher. As the temperature increased, the elastic modulus decreased, and the material was more prone to generate the stress relaxation.

The viscoelastic behavior of the bamboo fiber was investigated in this study. Bamboo fibers have the advantages of low density, light texture, low energy consumption, and biodegradability (Li *et al*. 2009; Reddy and Yang 2009; Ramires *et al*. 2010). The growing rate of bamboo is very fast. Its mechanical strengths are excellent (Abdullah Siam *et al.* 2019). The cost is relatively low. The toughness and tensile strength are good. Based on the above features, bamboo fiber is one of the best candidates as the reinforcement for green composites.

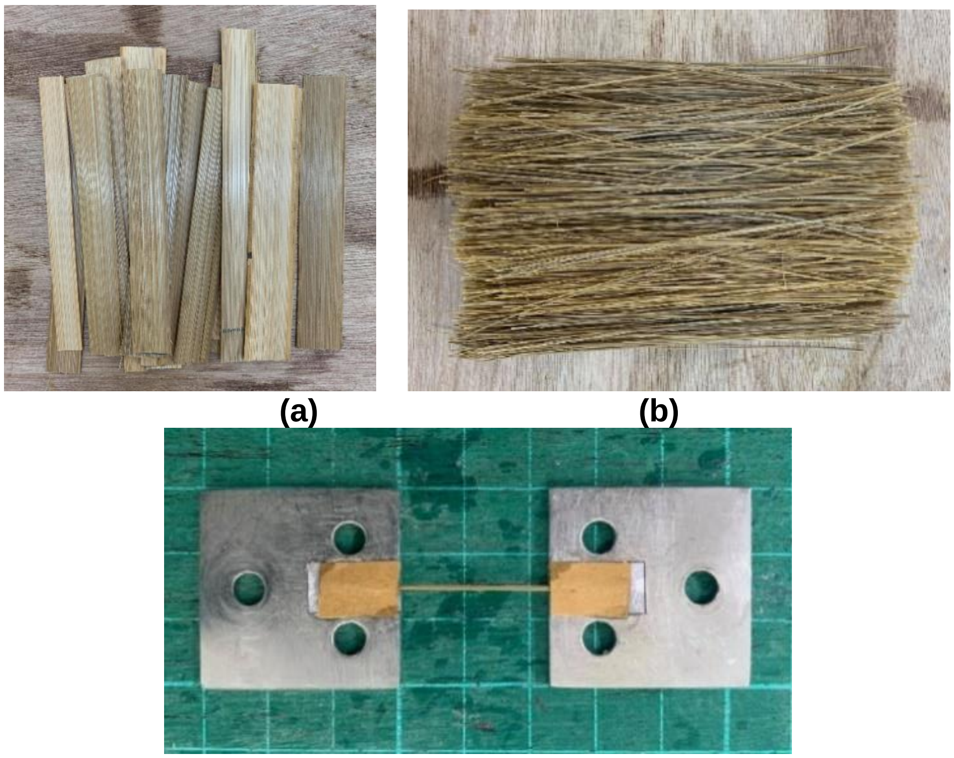
The obvious disadvantage of natural fibers as reinforcements for composites is the weak interfacial adhesion with the organic matrix. Bamboo fiber suffers the same problem of weak adhesion to the resin in the bamboo fiber reinforced polymer composites. To improve the adhesion between bamboo fiber and matrix material, the bonding to the matrix can be improved by alkali treatment (Takagi and Ichihara 2004; Kushwaha and Kumar 2010; Manalo *et al*. 2015). Osorio *et al*. (2011) reported that as it is treated with 1% to 3% concentration of sodium hydroxide solution, the strength of bamboo fiber increases slightly. The reason might be that, after undergoing alkali treatment, the weaker components (non-cellulose materials) of the fiber are removed, resulting in improved mechanical strength. Manalo *et al*. (2015) indicated that the composite whose bamboo fibers had undergone alkali treatment had stronger mechanical strength because the bonding force between fibers and polyester resin was increased.

Since the bamboo fiber can be used in the natural fiber reinforced composites, it is important to study the mechanical behavior of the single bamboo fiber. In the past, most research on bamboo fiber has been focused on elastic mechanical characteristics, and there has been less research on its viscoelastic properties. This study examined thorny bamboo; relaxation tests were conducted to observe the relaxation behavior of the bamboo fiber under various test conditions. The results suggested a nonlinear viscoelastic constitutive model to fit the relaxation curves, which could model the stress relaxation behavior of a bamboo fiber under different temperature or moisture content conditions in the studying range.

EXPERIMENTAL

Material Preparation

Bamboo fibers were isolated from thorny bamboo by the mechanical method. The bamboo culm was cut into slices with a length of 150 mm and a thickness of 0.8 mm without nodes with use of a filming cutting machine. Bamboo slices were thinned out to 0.55 mm in thickness with a utility knife, as shown in Fig. 1(a). Finally, bamboo fibers were cut from the bamboo slices along the fiber direction. The size of the bamboo fiber was 150 mm (L) × 0.5 mm (W) × 0.55 mm (H), as shown in Fig. 1(b). The equivalent diameter was 600 to 700 μm. Bamboo fibers were oven-dried at 80 °C for 3 h to remove the moisture.



(a) (b) (c)

Fig. 1. **(a)** Bamboo slices; **(b)** bamboo fibers; **(c)** specimen

For alkali treatment, dry bamboo fibers were soaked in 5 wt% sodium hydroxide solution at room temperature for 2 h, and the solution was stirred every 30 min. The weight ratio of bamboo fibers to sodium hydroxide solution was 1:30. After 2 h, alkaline treated bamboo fibers were washed with water and then oven-dried at 80 °C for 6 h.

Relaxation Test

Bamboo fibers were cut into 4.5 cm in length, and the average area of the cross section was approximately 0.235 mm^2. For the test specimen, the two ends of a single fiber were glued between sandpapers and aluminum fixtures, as shown in Fig. 1(c). The sandpaper was used to prevent the damage from the bamboo fiber during the clamping and to enhance the adhesion to the fixture. The gauge length of the sample was 2.2 cm, and five samples were tested for each case. The tensile tests were conducted at a speed of 0.3 mm/min to minimize the dynamic effect. The tensile tests were performed on a micro tensile test machine (a homemade model with installed load cell and optical ruler). The micro tensile test machine has a maximum load of 500 N and is installed with an optical ruler for measuring the displacement.

Bamboo fibers in both dry or wet conditions were used for the relaxation tests. For the wet condition, the moisture content was set to 0% or 60%, and the tests were conducted at room temperature. For the dry condition, the relaxation tests were conducted at 100 °C and 140 °C. As the bamboo fiber sample was clamped on the testing machine, the ring heater was installed around the fiber to control the temperature. The applied strains for relaxation tests were set to 0.2%, 0.4%, 0.6%, or 0.8% and maintained for 50 min. During the relaxation tests, the stress variations of the bamboo fiber were recorded.

Relaxation tests with multi-stage strain was performed. The original strain was 0.2% and maintained for 10 min. After 10 min, the strain increased by 0.2% and maintained for the same time. The process was repeated until the total strain reached 0.8%.

Relaxation Model

For the relaxation fitting model, a set of nonlinear empirical mathematical equations were used to fit the stress relaxation based on the experimental data. The modified Prony series model (Barbero 2011) was used to fit the relaxation stress. The parameters of fitted model were obtained by using a minimization algorithm to minimize the error between the modeled and experimental data values. The mathematical model for fitting the stress relaxation of the bamboo fiber is written as follows,

$$\sigma(t) = E_0\varepsilon_0 - (\mu_r\varepsilon_0)^n[1 - e^{\frac{-t}{t_\tau}}] \tag{1}$$

where t is the time, E_0 is the tangent modulus, ε_0 is the applied constant strain, μ_r and n are the material constants, and t_τ is the relaxation time.

The relaxation model was constructed based on the assumption that the applied strain was kept at a constant value. If the applied load was a variable strain with respect time, the stress can be derived by integrating with respect to the infinitesimal strain. By the superposition of the relaxation stress, a series of strain ε_i was superimposed to evaluate the relaxation of the stress as follows:

$$\begin{aligned}\sigma(t) &= \int_0^t (E_0 - \mu_r^n\varepsilon^{n-1}[1-e^{-\frac{t-\tau}{t_\tau}}])\frac{d\varepsilon(\tau)}{d\tau}d\tau \\ &= \int_0^t (E_0 - \mu_r^n\varepsilon^{n-1}[1-e^{-\frac{t-\tau}{t_\tau}}])d\varepsilon\end{aligned} \tag{2}$$

$$\sigma(t) = \sum_{i=0}^{m} (E_0 - \mu_r^n\varepsilon^{n-1}[1-e^{-\frac{t-\tau_i}{t_\tau}}])\Delta\varepsilon(\tau_i) \tag{3}$$

RESULTS AND DISCUSSION

Effect of Temperature on Relaxation

Figure 2 shows the stress relaxation curves of dry bamboo fibers at 100 and 140 °C for different applied strains. The relaxation curves were derived by averaging the data of 4 to 6 samples. For the strain of 0.2%, the stress relaxed completely to zero within 16 min at 100 °C, while it relaxed in 9 min at 140 °C. As the applied strain increased to 0.4%, there was a residual stress after relaxing for 50 min at 100 °C. However, the stress relaxed completely in 19 min at 140 °C. When the strain was 0.6%, there were residual stresses for both the cases at 100 and 140 °C. For the strain of 0.8%, the residual stress increased correspondently after relaxing for 50 min at 100 and 140 °C. Thus, the temperature had strong effect on the relaxation of the bamboo fiber. A higher temperature caused a higher degree of stress relaxation.

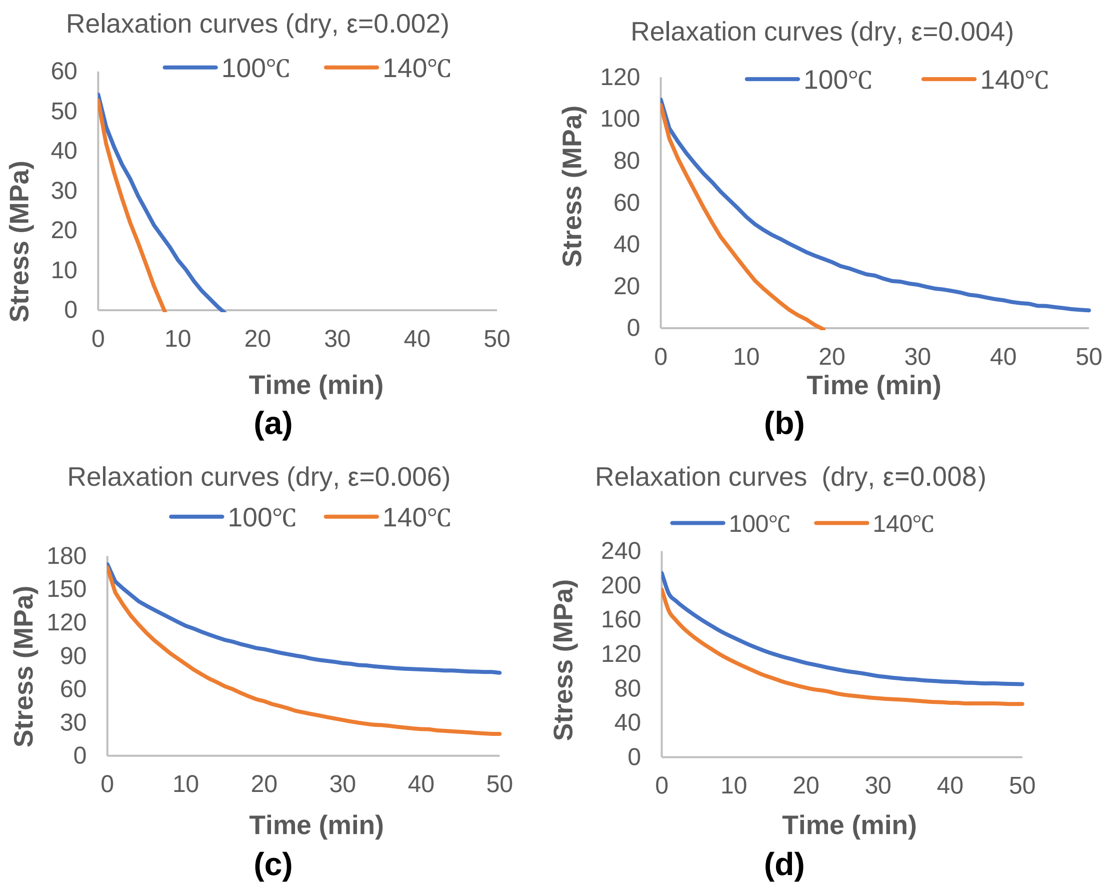


Fig. 2. Stress relaxation curves at 100 °C and 140 °C (a) ε=0.002, (b) ε=0.004, (c) ε=0.006, (d) ε=0.008

Figures 3 and 4 show the fitted and experimental stress relaxation curves of dry bamboo fibers at 100 and 140 °C, where the symbols are the average experimental stresses and the solid line is the fitted stresses by Eq. 1. The fitted parameters of Eq. 1 are shown in Table 1. The deviation was lower under the small strain, such as 0.2% and 0.4%. The tangent modulus, E_0, and relaxation time, t_τ, depended on the temperature, while μ_r and n were independent of the temperature.

Table 1. Fitted Parameter Values of Relaxation Equation (dry, 100 °C/140 °C)

T (°C)	E_0 (MPa)	t_τ (min)	μ_r $(\mathrm{MPa})^{\frac{1}{n}}$	n
100	27498	20.88	$4.98E+12$	0.2024
140	26800	9.18		
Remark	$\varepsilon_0 = 0.002$、0.004、0.006、0.008			

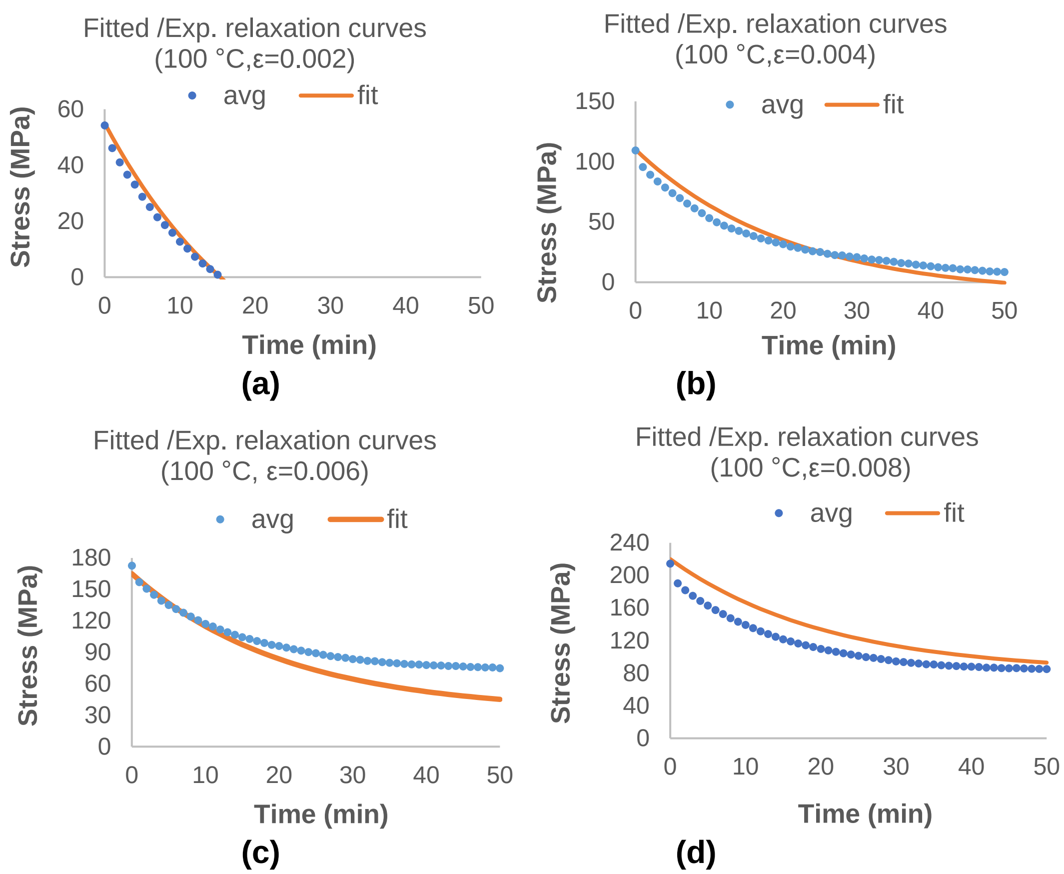
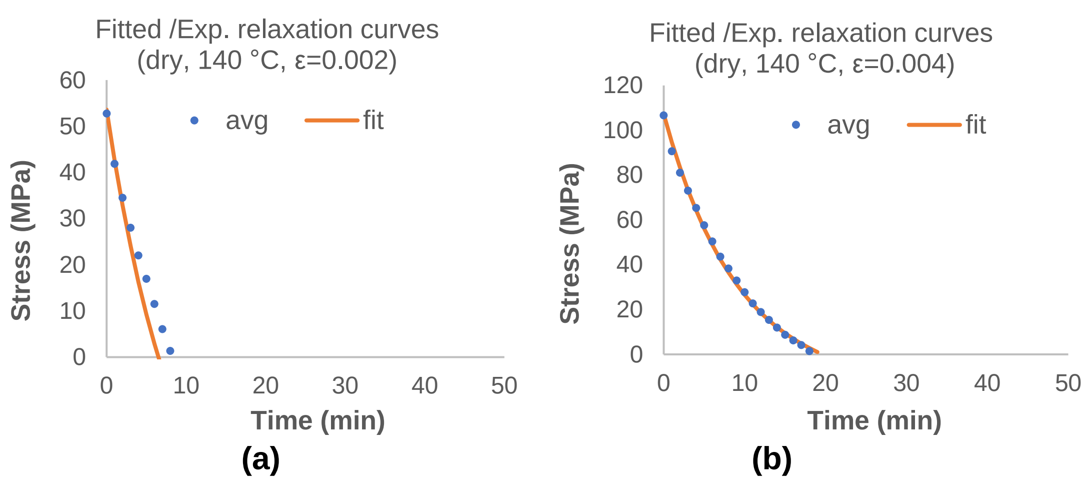
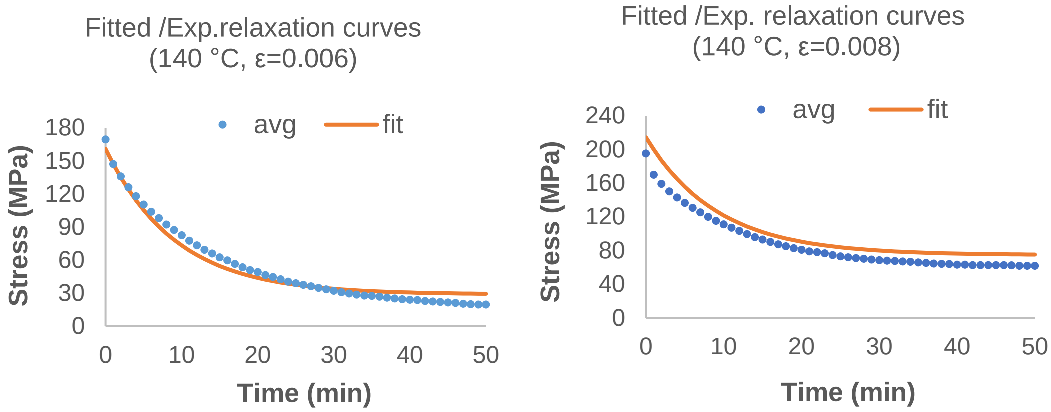


Fig. 3. Fitted and experimental stress relaxation curves at 100 °C (a) ε=0.002, (b) ε=0.004, (c) ε=0.006, (d) ε=0.008





(c) (d)

Fig. 4. Fitted and experimental stress relaxation curves at 140 °C (a) ε=0.002, (b) ε=0.004, (c) ε=0.006, (d) ε=0.008

To further integrate the temperature and time effect, the time–temperature superposition principle was applied, wherein a modified time scale ξ is related to the Williams-Landel-Ferry shift function as follows,

$$\xi(t) = \int_0^t \Phi[T(t')]dt' \tag{4}$$

$$\log \Phi = \frac{c_1(T - T_r)}{(c_2 + T - T_r)} \tag{5}$$

where T_r is 100 ℃, c_1 is -1.026, and c_2 is 75 ℃ by fitting the data in Table 1. This superposition principle is used to determine temperature-dependent mechanical properties of viscoelastic materials from known properties at a reference temperature. Equation 1 can be written as function of modified time as shown in Eq. 6.

$$\sigma(t) = E_0\varepsilon_0 - (\mu_r\varepsilon_0)^n[1 - e^{\frac{-\xi}{t_\tau}}] \tag{6}$$

Notice that there was no relaxation effect at the room temperature for Eq. (6) due to its small effect, as discussed later. Because the tangent modulus is also dependent on temperature, it can be written as follows,

$$E_0 = E_r - Be^{-\frac{T_1}{T - T_m}} \tag{7}$$

where E_r is the tangent modulus equaling 29.48 GPa at the room temperature, T_m = 25 ℃. By fitting the data in Table 1, B is 4.728 GPa and T_1 is 62.27 ℃. The relaxation of the bamboo fiber under the thermal effect can also be written as shown below,

$$\sigma(t) = E_0\varepsilon_0 - \Delta\sigma_T \tag{8}$$

$$\Delta\sigma_T = (\mu_r\varepsilon_0)^n[1 - e^{-\frac{-\xi}{t_\tau}}] \tag{9}$$

where $\Delta\sigma_T$ is the relaxation stress due to the thermal effect.

Effect of Moisture on Relaxation

Figure 5 shows the experimental and fitted stress relaxation curves of 60% wet bamboo fibers at a room temperature of 25 °C. In this case, only the hygro effect was considered, and the test environment was at room temperature. The relaxation effect was smaller under the hygro condition as compared than the thermal condition. For the strain of 0.002, the stress was not relaxed completely for 60% moisture content. The same trend was observed for the strains of 0.004 and 0.006. The residual stress was 70 to 75% of the original stress. The results suggested that the wet bamboo fiber cannot be performed to the exact shape at the room temperature as reported in literature (Chiu and Young 2020). As the wet bamboo fiber is deformed, there is a large residual stress retained after the preforming force is removed. This residual stress will cause large spring back after the preforming bamboo fiber is removed from the tool (Chiu and Young 2020a).

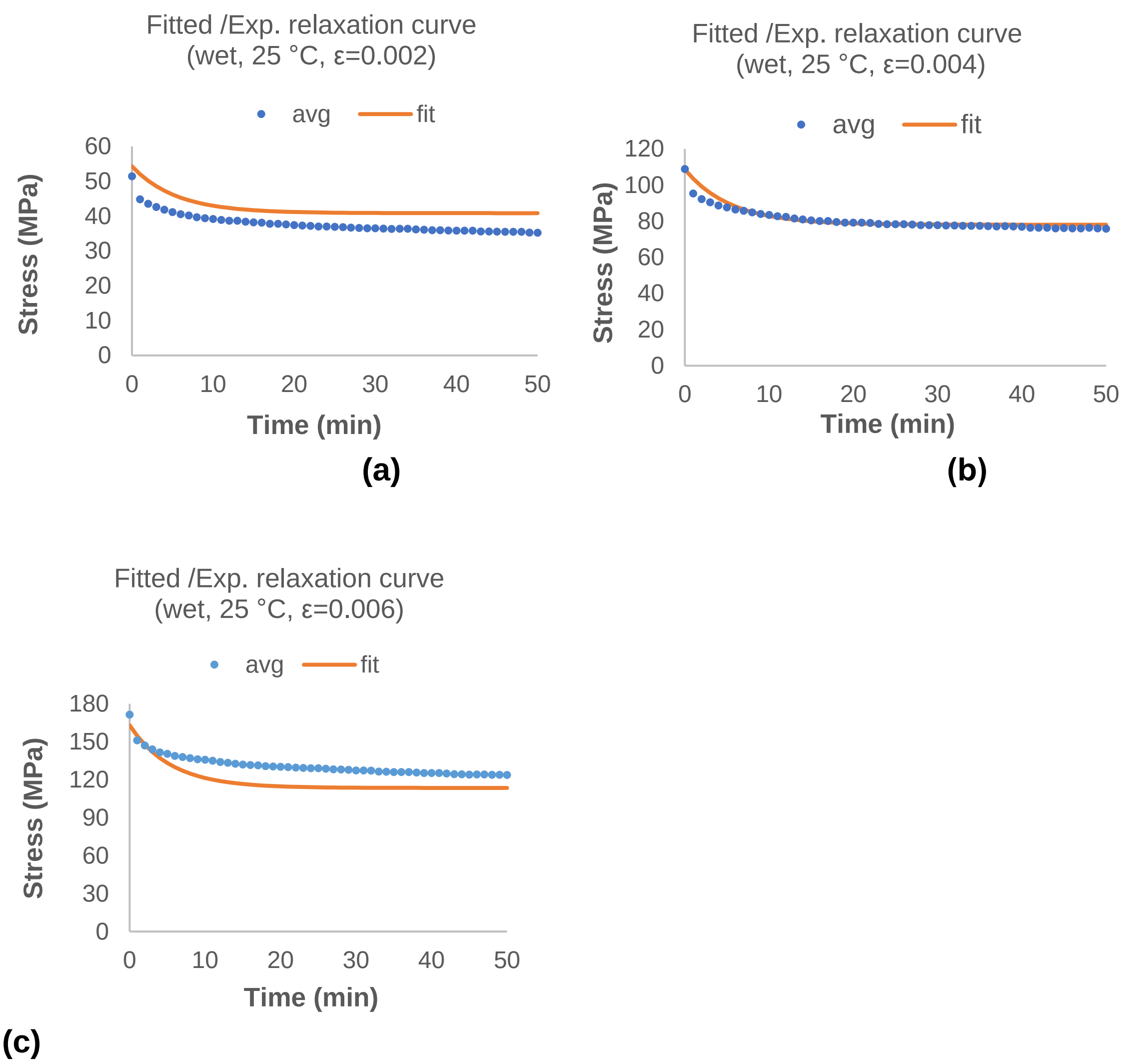


Fig. 5. Stress relaxation curves under moisture content of 60% at 25 °C for (a) ε=0.002, (b) ε=0.004, and (c) ε=0.006

Under the condition of 60% moisture content at 25 ℃, Eq. 1 can be used to model the stress relaxation behavior, which is expressed as follows.

$$\sigma(t) = E_0\varepsilon_0 - \Delta\sigma_h \tag{10}$$

$$\Delta\sigma_h = (\mu_r\varepsilon_0)^n[1 - e^{-\frac{t}{t_\tau}}] \tag{11}$$

The fitted parameter values of the equation are shown in Table 2. The parameters are different from those in the model for thermal effect. The fitted curves did not coincide completely with the experimental values, but the overall stress relaxation trends were consistent with the experimental values. The fitted relaxation curves can preliminarily present the stress relaxation variation of wet bamboo fibers (60% moisture content) at 25 ℃.

Table 2. Fitted Parameter Values of Relaxation Equation (wet, 25 °C)

E_0 (MPa)	t_τ (min)	μ_r $(\mathrm{MPa})^{\frac{1}{n}}$	n
27519	5.43	4492	1.184
Remark	$\varepsilon_0 = 0.002$、0.004、0.006		

Multi-stage Relaxation

The dry bamboo fiber at room temperature was assumed to have small viscous effect in the previous discussion. Because the relaxation of a dry bamboo fiber at a room temperature was quite slow, a multi-stage stress relaxation was conducted for the bamboo fiber under this condition. To compare the hygro and thermal effects, the bamboo fiber under the wet-25 ℃ (60% moisture content) and dry-140 ℃ conditions were tested in the multi-stage relaxation. Figure 6 shows the stress relaxation curves under multi-stage strain with five tests for each condition.

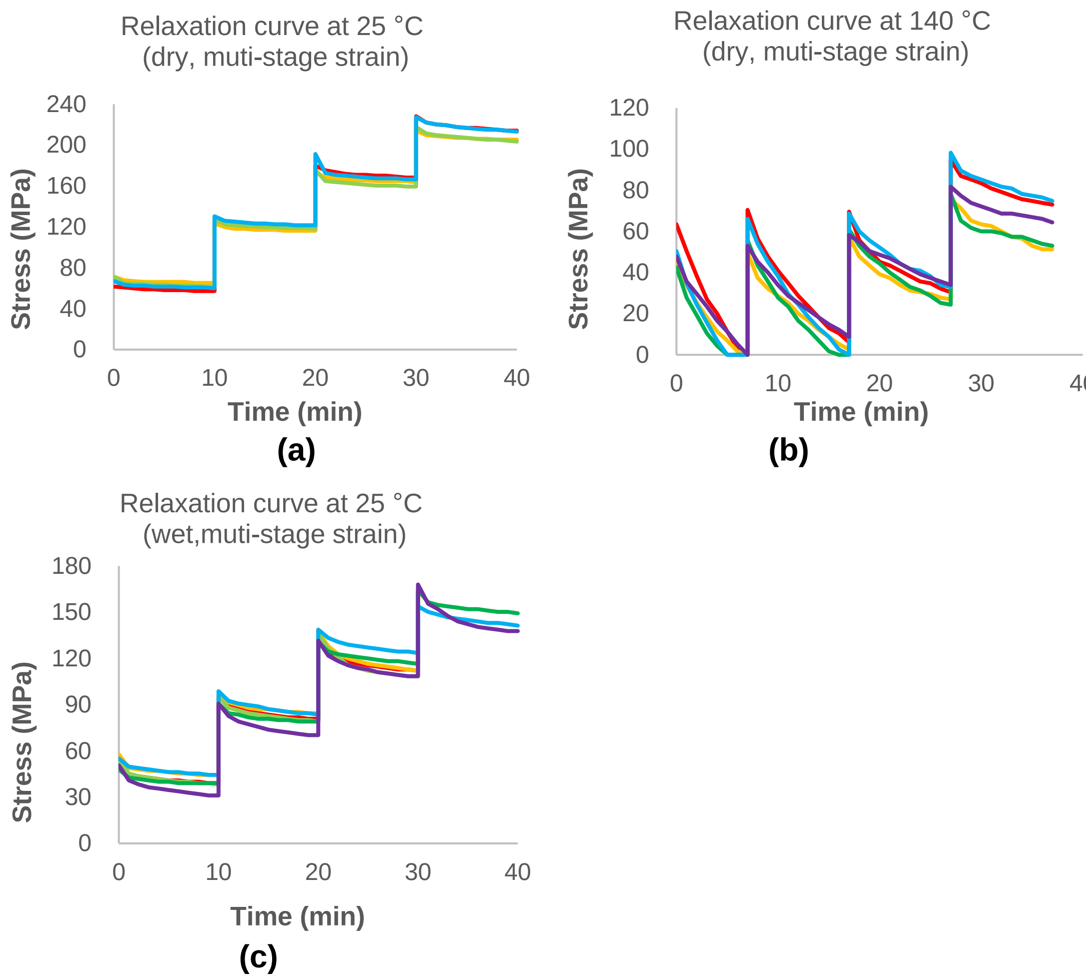


Fig. 6. Stress relaxation curves under multi-stage strain (a) 25 °C C, dry; (b) 140 °C C, dry; (c) 25 °C C, wet. Several tests were performed, as shown with different line colors.

At each stage, both the dry bamboo fiber and wet bamboo fiber at room temperature did not relax completely within 10 min. The relaxation amount for the dry fiber in the room temperature was relatively small. Furthermore, the relaxation range of the dry bamboo fiber at a high temperature was quite large, and the stress decreased to zero at the first and second stages of 0.2% applied strain. In conclusion, the moisture content and temperature are the important factors affecting the stress relaxation behavior, and the stress relaxation phenomenon may be greater in the hot and humid environment. However, due to the limitation of the current equipment, the temperature and moisture content of a bamboo fiber cannot be well controlled at the same time. The experiments in both the thermal and hygro conditions might be expected in a future study.

Multi-stage Creep

To verify the relaxation model discussed in the previous section, the relaxation model was used to calculate the creep behavior and compared with the experimental results. A multi-stage creep test was conducted for the wet bamboo fiber under 60% moisture content and room temperature. An initial 20 N load was applied on one end of a wet bamboo fiber with the other end fixed. After 10 min, the applied load was increased to

25 N by adding another 5 N, which was held for another 10 min. The load was increased 5 N for every 10 min until the fiber was fractured.

The relaxation model was based on the assumption that the applied strain was a constant, and the stress varied with time. For the creep test, the applied stress was a constant, and the strain might change with time. Therefore, for using the relaxation mode to calculate the creep behavior, the applied strain must increase with to ensure a constant stress. Then, Eq. 10 may be written as follows.

$$\sigma(t)=\sum_{i=0}^{m}(E_0\Delta\varepsilon_i-\Delta\sigma_i(t)) \tag{12}$$

$$\Delta\sigma_i(t)=\mu_r^n\varepsilon^{n-1}[1-e^{-\frac{t-i\Delta t}{t_\tau}}]\Delta\varepsilon_i \tag{13}$$

The time was discretized to $i\Delta t$ for the calculation for the stain variation, and Δt is the time increment. A series of strain $\Delta\varepsilon_i$ was superimposed to keep the stress equaling the applied load in Eq. 12. For every time step, a new strain increment, $\Delta\varepsilon_i$, must be applied to compensate the decrease of the stress due to relaxation. The resulting total strain will be the strain in the creep test.

Figure 7 shows the modeled and experimental strain for the multi-stage creep test of the wet bamboo fiber, where five experimental data were shown. The parameters for the relaxation model are shown in Table 2 for the bamboo fiber under wet and room temperature conditions. There was some deviation between the modeled and experimental values, but the creep response of wet bamboo fiber under multi-stage load was adequately modeled by the relaxation equation. The result indicated that the relaxation and creep properties were compatible, which means that the relaxation fitting model can be used to model the viscoelastic behavior of the bamboo fiber to some extent.

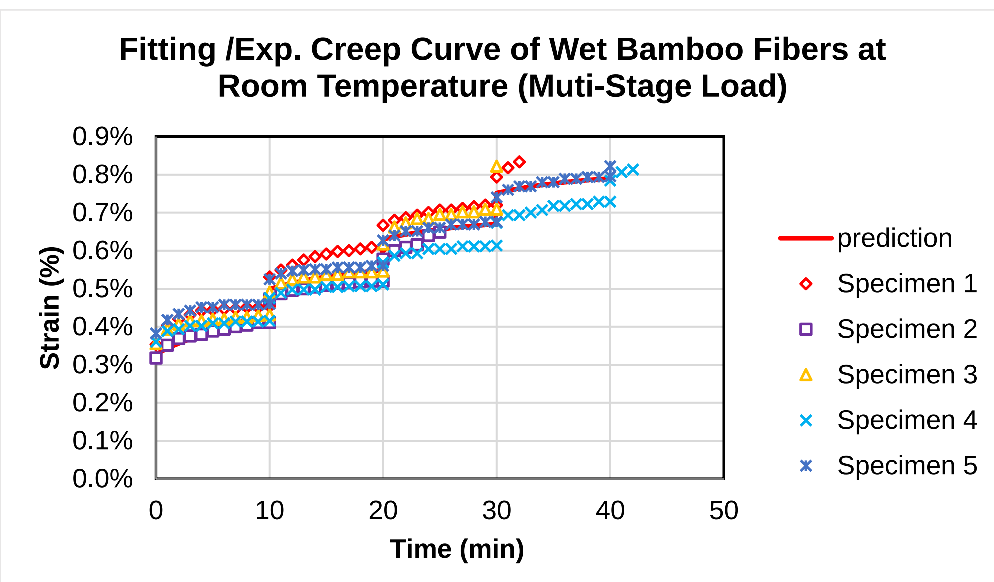


Fig. 7. Modeled and experimental creep strain under multi-stage load (relaxation model)

CONCLUSIONS

1. The bamboo fiber exhibited viscoelastic behavior under both hygro and thermal conditions. High moisture content and high temperature accelerate the relaxation rate and increase the amount of relaxation stress. The temperature was the most important factor for viscoelasticity.
2. The relaxation of the bamboo fiber depends on the time and the amount of the applied strain. For small strain, the stress may relax to zero within 10 min under the high temperature condition for the bamboo fiber.
3. A nonlinear viscoelastic model was proposed to model the relaxation behavior of the bamboo fiber under the thermal and hygro conditions respectively. The model was used to model the creep behavior of the bamboo fiber and gave acceptable results as compared to the experimental data.

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