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Effect of Thermal Aging on Dynamic Mechanical Performance of a Novel Structural Adhesive

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ABSTRACT

This study mainly evaluates the thermal aging properties of a new room temperature cured structural adhesive. Multiple adhesive specimens were thermal aged at 80℃, 100℃, 120℃ and 140℃, respectively, and subjected to the dynamic mechanical analysis (DMA) at different temperatures and frequencies. According to the results of DMA and thermodynamic analysis, the performance of the adhesive changes little after aging at 80℃, 100℃ and 120℃ for 30 days. However, the adhesive performance became unstable at 140℃, and deteriorated rapidly with the extension of aging time. Hence, this structural adhesive is suitable for the environment below 120℃. In addition, the generalized curve of the structural adhesive aging for 20 days at 100℃ was obtained, following the principles of time-temperature equivalence superposition and time-aging time equivalence superposition. The test time was shortened significantly. This research provides an accelerated characterization strategy for the long-term mechanical properties of other polymers, making it possible to obtain the generalized curve of polymers aging for a specific time at a specific temperature.

1. INTRODUCTION

With excellent mechanical properties and superior environmental stability, building structural adhesives have gained popularity in the engineering field, and realized wide application in the bonding and reinforcement of structural components of buildings and in the reconstruction of existing buildings [1-3]. When applied to load-bearing structural components of buildings, structural adhesives are exposed to a complex environment for a long period, and disturbed by various external factors, such as temperature, humidity, and ultraviolet light. These factors work together to cause a series of macro and micro changes to the structure and composition of the structural adhesives [4-6], which in turn bring about aging failure, resource waste, and safety accidents. Therefore, the aging of building structural adhesives in various service environments cannot be ignored.

In the past decades, the thermal aging of structural adhesives has been extensively studied both theoretically and experimentally, as temperature is considered a key factor in the aging process. Researchers agree that a high temperature can quickly improve the effect of structural adhesives [7-10]. Therefore, a short test period, several days to tens of days, is often selected for the thermal aging test on structural adhesives at different temperature levels [11-13]. But the short test period cannot reflect the true thermal aging performance of structural adhesives objectively in the long run, for buildings often serve for decades or centuries. Besides, the test period is far shorter than the test time specified in relevant technical codes [14, 15].

The principle of time-temperature equivalence superposition provides researchers with an effective tool to acquire test data in a long time range, under limited test period and conditions. This principle has been widely adopted to quickly predict the long-term mechanical properties of structural adhesives [16-20]. For instance, many researchers have applied the principle to analyze polymer performance after thermal aging test, focusing mostly on static mechanical properties. However, it is the dynamic mechanical properties that truly reflect the working performance of structural adhesives under service conditions. This is because structural adhesives are typical time-dependent viscoelastic materials, and buildings need to bear dynamic loads for a long time.

Dynamic mechanical analysis (DMA) provides a desirable tool to explore the viscoelastic behavior of polymers [21-23]. Through the DMA, it is possible to obtain the polymer features at different temperatures and frequencies in the test range [24-27]. The representative polymer features, including transition temperature, storage modulus, and loss modulus, directly mirror the strength and durability of materials.

This paper probes into the effect of thermal aging on a new building structure adhesive cured at room temperature, and characterizes its dynamic mechanical properties before and after thermal aging in a systematic manner. Considering the limitation of data collected from the short test period, the principles of time-temperature equivalence superposition and time-aging time equivalence superposition were employed to accelerate the characterization of the test data, which effectively shorten the test cycle. Our research sheds light on how to obtain the generalized curve of aged polymers at a specific temperature for a specific time, providing a reference for the accelerated characterization of long-term mechanical properties of other polymers.

2. METHODOLOGY

2.1 Materials and samples

Our structural adhesive was prepared from epoxy resin (E51), Novolac epoxy resin (F51), Qishi toughening agent, and m-Xylylenediamine (m-XDA) hardener.

Firstly, 50g E50 epoxy resin was mixed with 50g Novolac F51 epoxy resin at a ratio of 1:1 to obtain a 100g matrix of the adhesive resin. Considering the high viscosity of F51 at room temperature, the two resins were heated and mechanically stirred for 2 hours to ensure the completeness and evenness of the mixture.

Next, 15g Qishi toughening agent and 18g m-Xylylenediamine hardener were added to every 100g of resin mixture. After being stirred for 10min, the mixture was relocated into a vacuum box to remove bubbles for 15min. Finally, the mixture was poured into the prepared mold, and the samples were cured at room temperature for seven days, waiting to be used in the subsequent test.

As shown in Figure 1, the sample size is 25mm × 5mm × 2.5mm.

2.2 Instruments and methods

The thermal aging test of our structural adhesive was carried out in a 401A thermal aging test chamber, while the thermal analysis test on dynamic mechanical properties utilized a DMA 8000 rheometer.

For the thermal aging test, four temperatures (80℃, 100℃, 120℃, and 140℃) were set in the range of $Tg \pm 20$℃, as the glass transition temperature (Tg) of the adhesive is 108℃. To ensure the test accuracy, the thermal aging box was set to the corresponding temperature the day before the first day of the test. As a result, the box had reached the designed thermal aging temperature, when the epoxy structural adhesive was put into the box.

Next, the samples, after being cured at room temperature for 7 days, were put into the thermal aging box. The test period at each thermal aging temperature was kept as 30 days. During the period, a batch of six samples were taken out every 5 days for the subsequent test on dynamic mechanical properties. The latter test aims to disclose the changes of adhesive properties.

In each batch, the temperature spectrum of three samples were scanned, while the frequency spectrum of the other three were scanned. The mean of each three samples was taken as the test result. The temperature scanning was performed in the single cantilever bending mode, with the temperature rising from room temperature to 150℃ at the rate of 2℃/min and the strain of 0.1%. The frequency scanning was performed in the same mode, at various temperature levels (40℃, 50℃, 60℃, 70℃, 80℃, 90℃ and 100℃). At each temperature level, the test frequency was increased from 0.01Hz to 100Hz, with the strain of 0.1%.

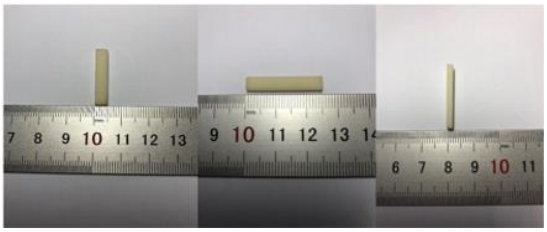
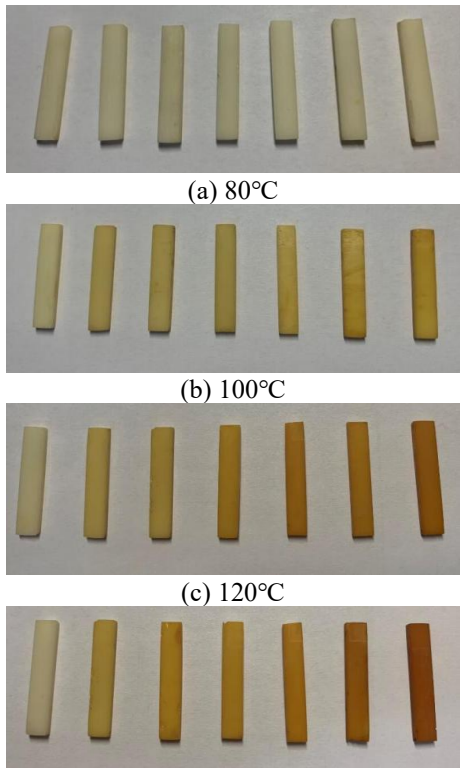


Figure 1. A test sample



(a) 80℃

(b) 100℃

(c) 120℃

(d) 140℃

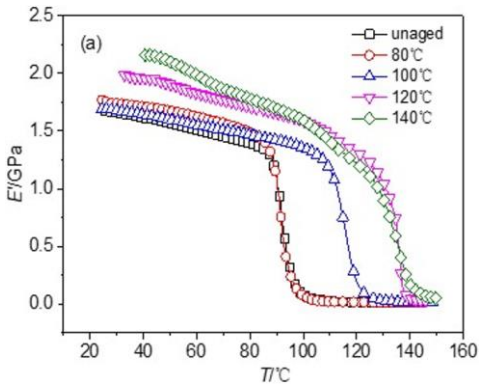
Figure 2. Samples at different aging temperature levels on different aging days

3. RESULTS AND ANALYSIS

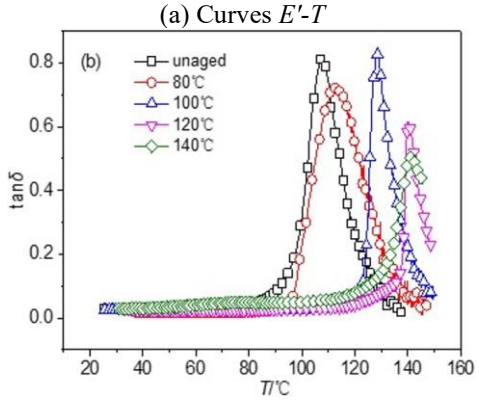
3.1 Temperature spectrum analysis

The structural adhesive samples were aged at four thermal aging temperatures (80℃, 100℃, 120℃, and 140℃) for 30 days. Six samples were taken out every 5 days (Figure 2). In each subgraph, the leftmost sample is the sample without thermal aging but cured at room temperature for 7 days. Excluding that sample, the remaining subgraphs are the samples cured for 0d, 5d, 10d, 15d, 20d, 25d, and 30d, respectively, from left to right. As shown in Figure 2(a), the adhesive color did not change greatly during the 30d aging period at 80℃. As shown in Figure 2(b)-(d), the adhesive gradually became darker, as the aging time extended during the 30d aging period at 100℃, 120℃ and 140℃. The higher the temperature, the deeper the color.

Figure 3 presents the storage modulus-temperature curves and loss tangent-temperature curves of the adhesive after aging for 30 days at different temperature levels. After the 30d aging at different temperatures, the storage modulus of the adhesive increased (Figure 3(a)), because the post-curing of adhesive through thermal aging increases the crosslinking degree of the adhesive and pushes up E'. Although the glass transition temperature of the adhesive increased with the aging temperature (Figure 3(a)), the curves are all temperature spectrum curves of standard cross-linked polymers in the glass state, glass transition state, and high elastic state, which correspond to the aging temperatures of 80℃, 100℃ and 120℃, respectively. When the aging temperature rose to 140℃, the initially high modulus of the adhesive declined with the rising test temperature, without exhibiting an obvious glass transition state. Thus, this epoxy structural adhesive should not be used at above 120℃.

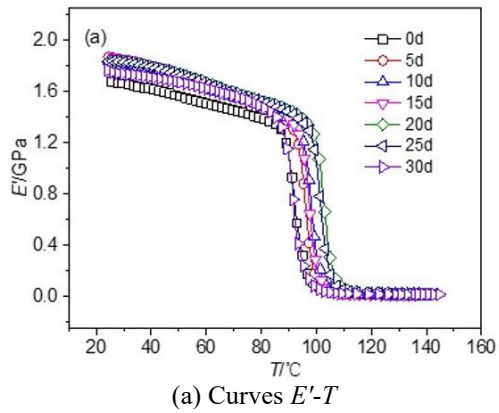


(a) Curves E'-T

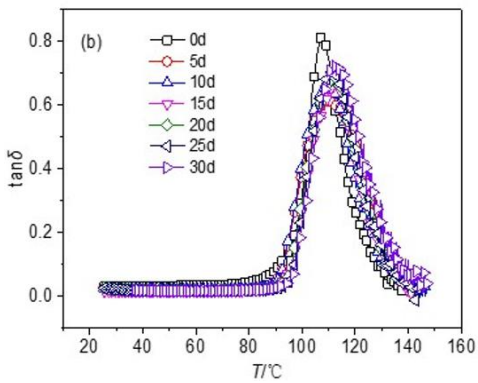


(b) Curves tanδ-T

Figure 3. Temperature curves at different temperature levels after 30 days of curing



(a) Curves E'-T



(b) Curves tanδ-T

Figure 4. Temperature scanning curve at 80℃ after 30 days

Table 1. *Tg* of the adhesive after different aging days at 80℃

	Unaged	Aging for 5 days	Aging for 10 days	Aging for 15 days	Aging for 20 days	Aging for 25 days	Aging for 30 days
Glass transition temperature *Tg*	108℃	109℃	110℃	111℃	112℃	113℃	113℃

Table 2. *Tg* of the adhesive after different aging days at 100℃

	Unaged	Aging for 5 days	Aging for 10 days	Aging for 15 days	Aging for 20 days	Aging for 25 days	Aging for 30 days
Glass transition temperature *Tg*	108℃	109℃	111℃	114℃	117℃	121℃	123℃

Table 3. *Tg* of the adhesive after different aging days at 120℃

	Unaged	Aging for 5 days	Aging for 10 days	Aging for 15 days	Aging for 20 days	Aging for 25 days	Aging for 30 days
Glass transition temperature *Tg*	108℃	126℃	130℃	131℃	133℃	136℃	142℃

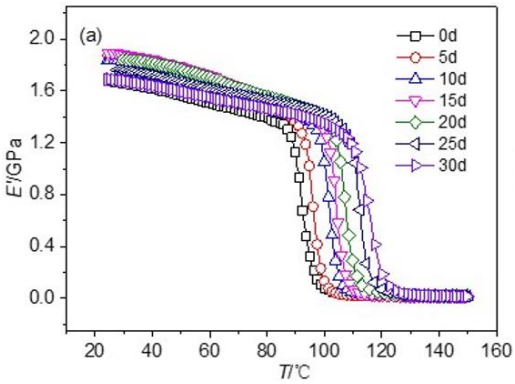
Table 4. *Tg* of the adhesive after different aging days at 140℃

	Unaged	Aging for 5 days	Aging for 10 days	Aging for 15 days	Aging for 20 days	Aging for 25 days	Aging for 30 days
Glass transition temperature *Tg*	108℃	131℃	135℃	139℃	141℃	145℃	143℃

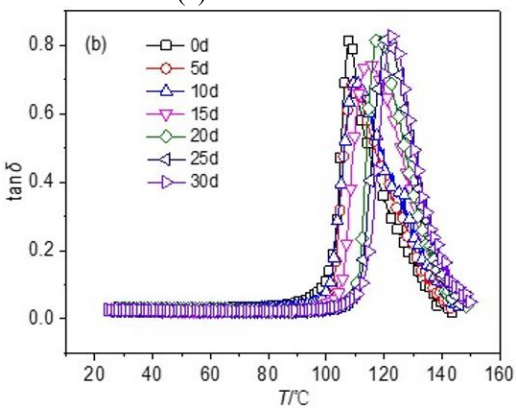
Figures 4-7 show the E'-T curves and tanδ-T curves of our structural adhesive after 30d aging at 80℃, 100℃, 120℃ and 140℃, respectively. The glass transition temperature *Tg* of different aging days under four different aging temperatures are listed in Tables 1-4, respectively. It can be observed that the storage modulus of the adhesive increased with the aging temperature; the E'-T curves moved to high temperature gradually, as the glass transition temperature rose continuously with the elapse of aging time (0-30 days).

At all four aging temperatures, the aged samples had a greater storage modulus E' than the unaged samples. The reason is that, during the post-curing of this room temperature curing system, the thermal aging condition increases the crosslinking degree and curing degree of the adhesive. On the one hand, in the temperature scanning mode, more molecular segments are excited, and the degree of crosslinking is increased, as the test temperature rises. On the other hand, the temperature growth increases the free volume of molecules in the adhesive, and expands the movable space of molecular segments, thus increasing the degree of crosslinking. That is why both the storage modulus of the adhesive and the glass transition temperature increased.

Note that the decline rate of the storage modulus increased significantly from 5 days to 30 days at the aging temperature of 140℃, indicating that the performance of adhesive decreases sharply at 140℃, as shown in Figure 7(a).

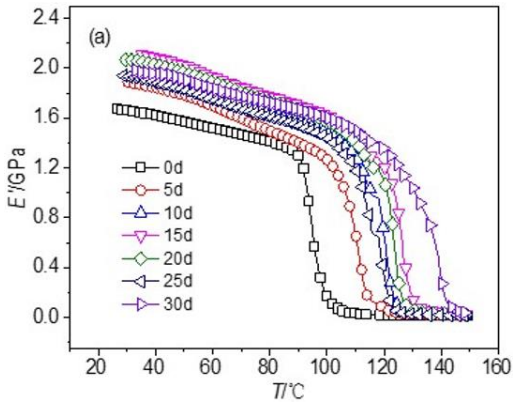


(a) Curves *E'*-*T*

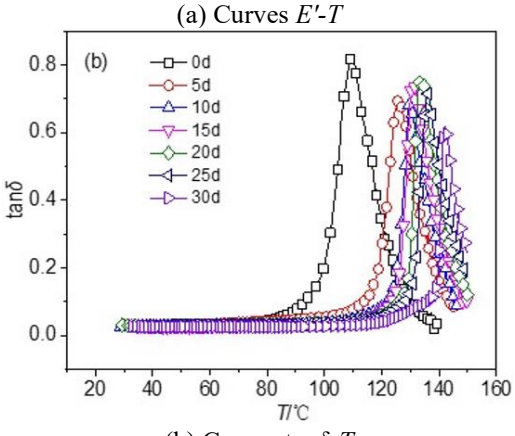


(b) Curves tanδ-*T*

Figure 5. Temperature scanning curve at 100℃ after 30 days

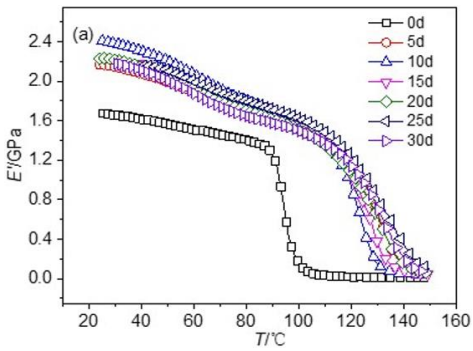


(a) Curves *E'*-*T*

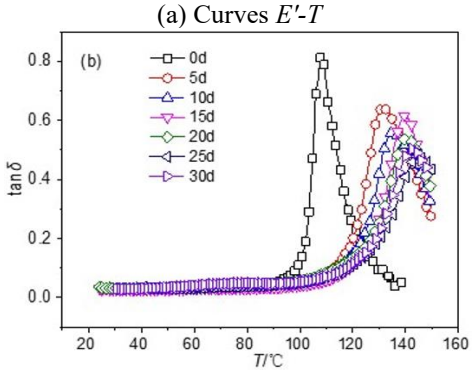


(b) Curves tanδ-*T*

Figure 6. Temperature scanning curve at 120°C after 30 days



(a) Curves *E'*-*T*



(b) Curves tanδ-*T*

Figure 7. Temperature scanning curve at 140°C after 30 days

3.2 Frequency spectrum analysis

Figures 8-11 show the storage modulus-frequency curves at four different aging temperatures, respectively. After logarithmic treatment, the storage modulus-frequency curves are approximately straight lines. Therefore, our adhesive is a simple thermorheological material, which can be characterized by accelerated characterization methods like the time-temperature equivalence principle.

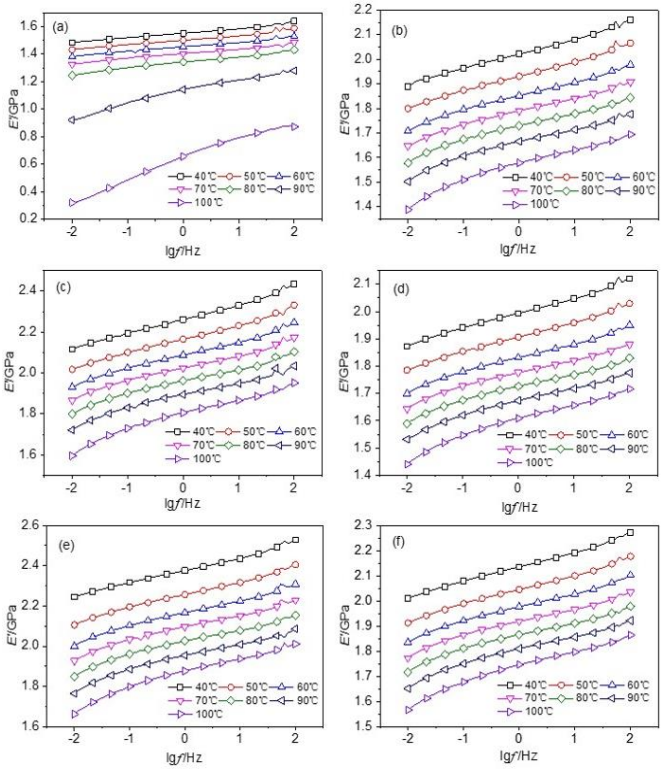


Figure 8. *E'*-lg*f* curves at 140℃ after different aging periods (a) Unaged sample, (b) Aging for 10 days, (c) Aging for 15 days, (d) Aging for 20 days, (e) Aging for 25 days, (f) Aging for 30 days

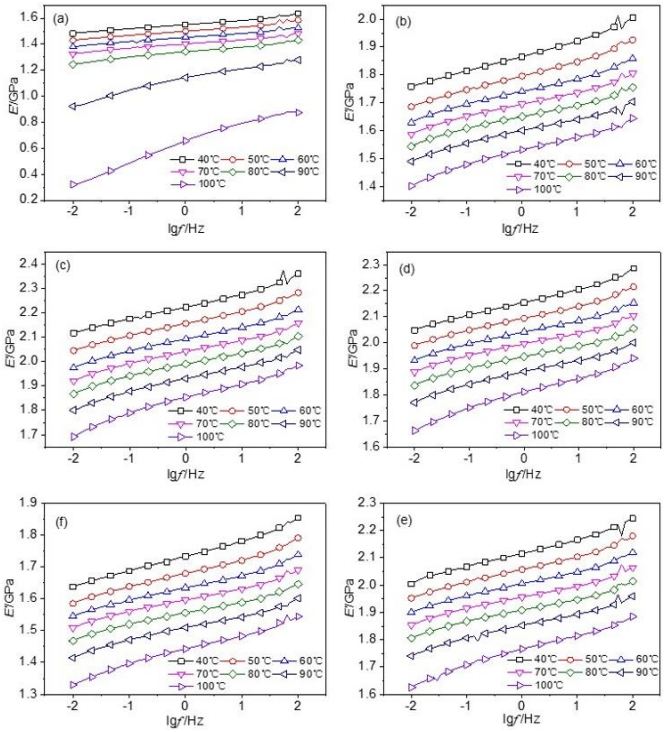


Figure 9. *E'*-lg*f* curves at140℃ after different aging days (a) Unaged sample, (b) Aging for 10 days, (c) Aging for 15 days, (d) Aging for 20 days, (e) Aging for 25 days, (f) Aging for 30 days

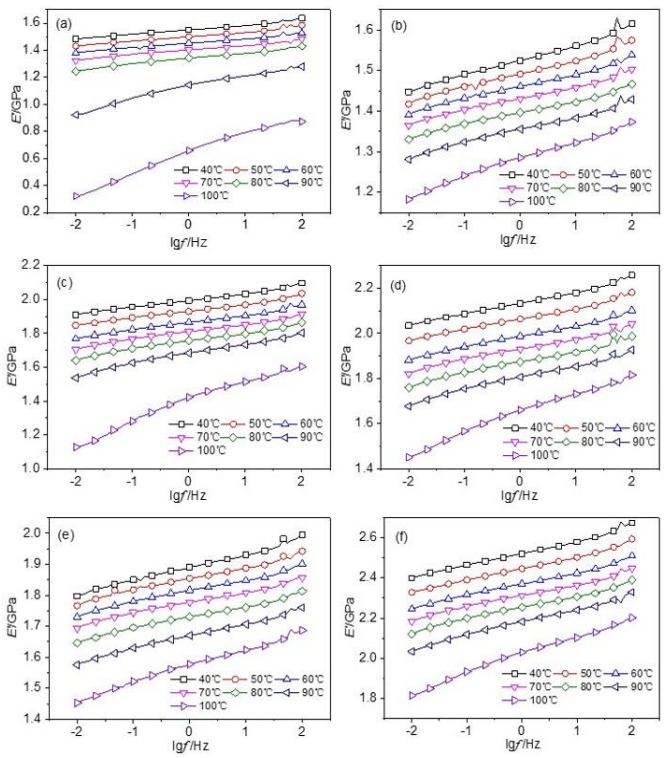


Figure 10. *E'*-lg*f* curves at100℃ after different aging days (a), Unaged sample (b) Aging for 10 days, (c) Aging for 15 days, (d) Aging for 20 days, (e) Aging for 25 days, (f) Aging for 30 days

As shown in Figures 8(a)-11(a), the unaged curve at 90℃ and 100℃ changed inconsistently with the curves at 40℃-80℃. With the growth of test frequency, the storage modulus changed by a small amplitude, and the storage modulus curves were quite gentle at 40℃-80℃. The change range increased significantly and the curve became steeper, after the test temperature reached 90℃ and 100℃.

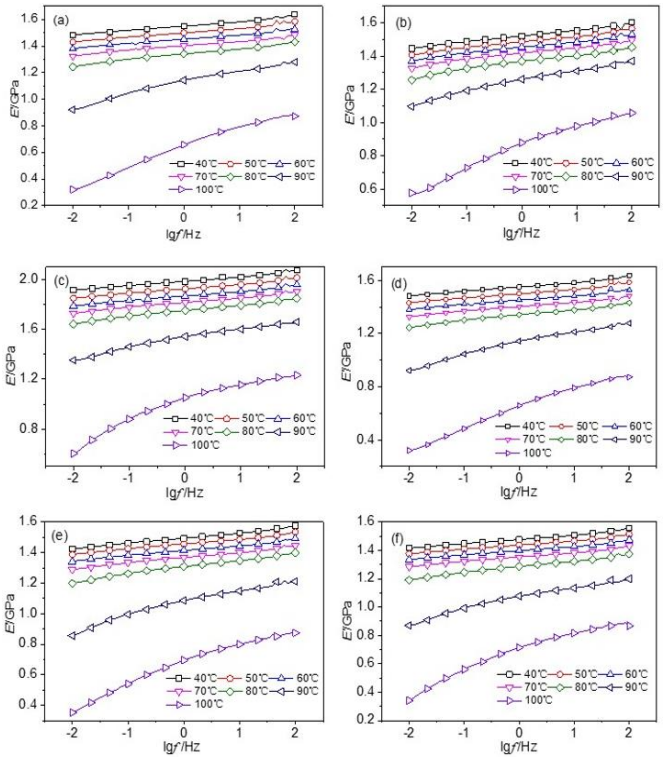


Figure 11. *E'*-lg*f* curves at 80℃ after different aging days (a) Unaged sample, (b) Aging for 10 days, (c) Aging for 15 days, (d) Aging for 20 days, (e) Aging for 25 days, (f) Aging for 30 days

Nevertheless, the change trend of storage modulus-frequency curves and the space of each curve remained consistent at seven test temperatures after 30 days of thermal aging at 140℃, 120℃ and 100℃ (Figures 8 (b)-(f) - 11(b)-(f)). A possible reason is that the motions of the internal molecular segments of the adhesive have reached an equilibrium state after high-temperature aging, and the segments make an equilibrium response with the uniform increase of the test temperature.

As shown in Figure 11, the storage modulus-frequency curves of aging samples obeyed the same change law as those of the unaged samples. This is because some molecular segments are not excited at 80℃, but are excited at a higher test temperature (90℃, or 100℃) with the steep curve.

Figures 12-15 show the loss tangent-frequency curves (tanδ-lg*f*) at four different aging temperatures, respectively. The curves after different aging days at each aging temperature exhibited the same change law as those in Figures 8-11. The variation pattern of the unaged curves at 90℃ and 100℃ were inconsistent with the curves at 40℃-80℃. At low test temperatures like 40℃, 50℃, 60℃, 70℃, and 80℃, the motions of the molecular chain segments “freeze”, due to the small internal friction, eliminating the need to overcome the friction caused by the migration between the segments. That explains the small tanδ, and the flat change of the tanδ-lg*f* curve.

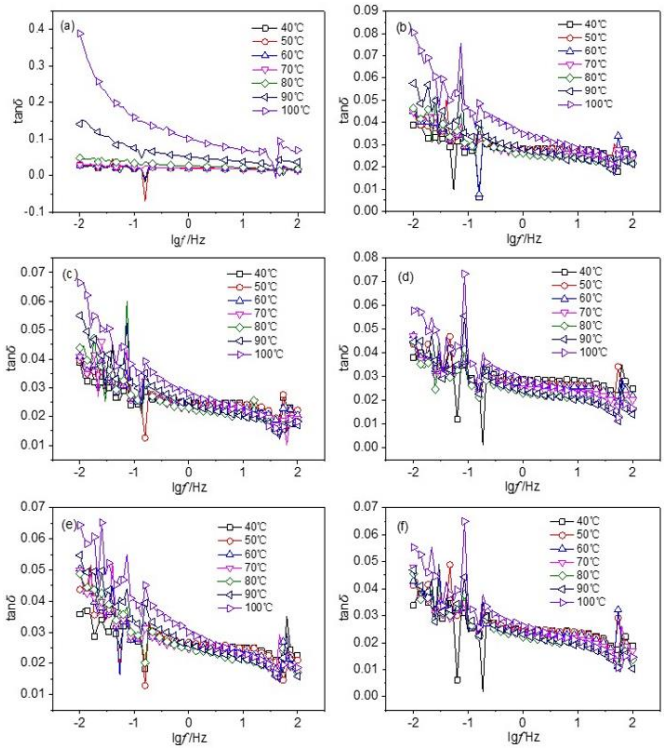


Figure 12. tanδ-lg$f$ curves at 140℃ after different aging days (a) Unaged sample, (b) Aging for 10 days, (c) Aging for 15 days, (d) Aging for 20 days, (e) Aging for 25 days, (f) Aging for 30 days

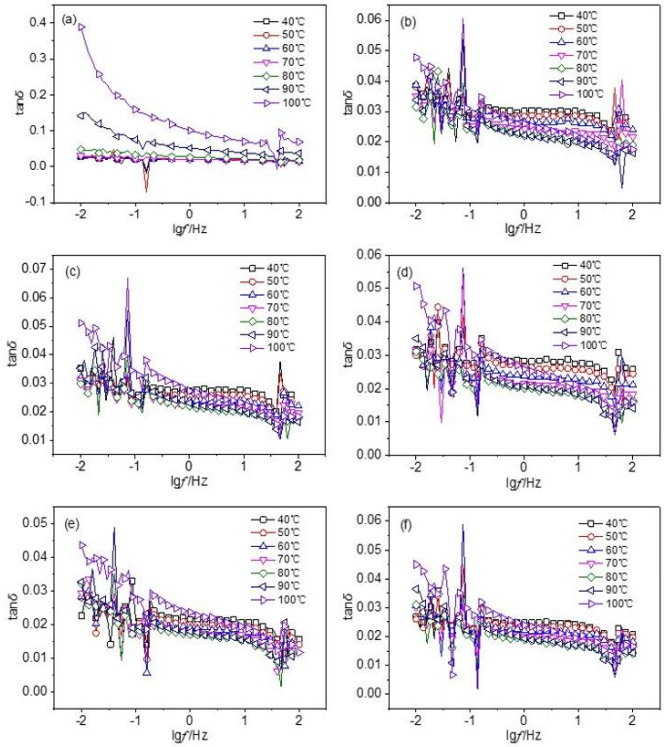


Figure 13. tanδ-lg$f$ curves at 120℃ after different aging days (a) Unaged sample, (b) Aging for 10 days, (c) Aging for 15 days, (d) Aging for 20 days, (e) Aging for 25 days, (f) Aging for 30 days

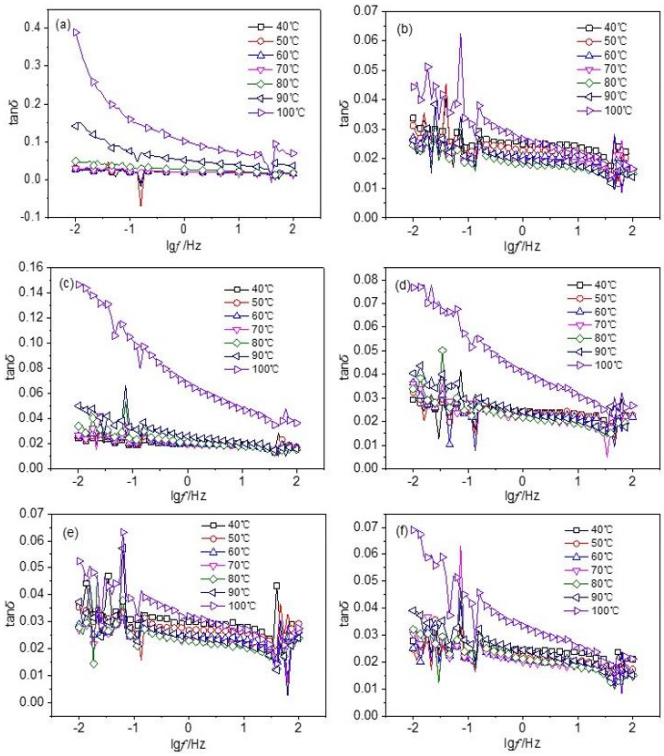


Figure 14. tanδ-lg$f$ curves at 100℃ after different aging days (a) Unaged sample, (b) Aging for 10 days, (c) Aging for 15 days, (d) Aging for 20 days, (e) Aging for 25 days, (f) Aging for 30 days

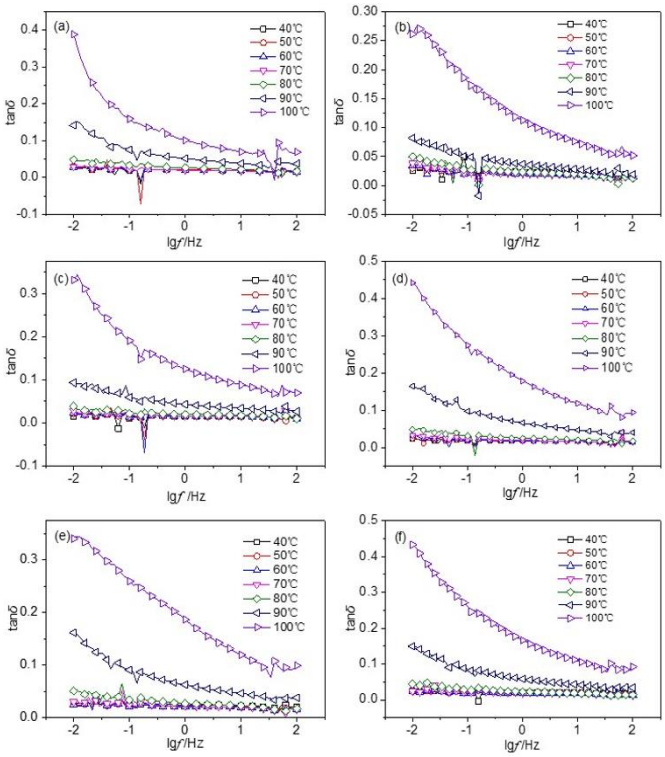


Figure 15. tanδ-lg$f$ curves at 80℃ after different aging days (a) Unaged sample, (b) Aging for 10 days, (c) Aging for 15 days, (d) Aging for 20 days, (e) Aging for 25 days, (f) Aging for 30 days

The "frozen" state of the molecular chain segments starts to end at 90℃ and 100℃. At this time, the segments have certain movement ability, but the large friction of the motions needs to be overcome. As a result, the internal friction and tanδ increase, and the change of the tanδ-lg$f$ curve intensifies with steep curve. However, there was not much difference between

the tanδ-lg$f$ curves after 30 days of thermal aging at 140℃, 120℃ and 100℃ at the seven test temperatures (Figures 12-14). As shown in Figure 11, after thermal aging at 80℃ for 30 days, the tanδ-lg$f$ curves of aging samples had basically the same change law as those of unaged samples. In addition, the value tanδ fluctuated, due to the resonance between the instrument and the test sample in the low and high test frequency bands (< 0.158Hz and > 65Hz).

4. ACCELERATED CHARACTERIZATION ANALYSIS

By analyzing the relaxation phenomenon of molecular segments, the same mechanical relaxation could be observed both in a short time at high temperatures, and in a long time at a low temperature. Consequently, the effect of temperature rise is equivalent to the extension of the observation time, hence the principle of time-temperature equivalence superposition.

Similarly, viscoelastic materials after different aging times have similar performance curves, thus the principle of time-aging time equivalence superposition. The superimposed master curve can be obtained by translating the viscoelastic curve of the reference aging time along the logarithmic time axis. In this way, the dynamic mechanical properties of the structural adhesive after thermal aging can be accelerated.

Firstly, the mechanical properties of our adhesive at different aging temperatures were accelerated by the principle of time-temperature equivalence superposition, with the test temperature as the reference temperature for accelerating characterization. As shown in Figure 16, T_a is the aging temperature, and T_n (n = 0, 1, 2, 3...) is the test temperature. The test temperature T_0 was taken as the reference temperature, and the generalized curves under the aging temperature T_a and test temperature T_0 were obtained by translation.

Combined with the test results, the principle of time-temperature equivalence superposition was employed to process Figure 11. The test data at aging temperature 100℃ were taken as an example (T_a = 100℃). The test temperature 100℃ was selected as the reference temperature (T_0 = 100℃), and the curves of other test temperatures were translated to the reference curve. The generalized curve at test temperature 100℃ for different days of aging at 100℃ was thus obtained (Figure 17).

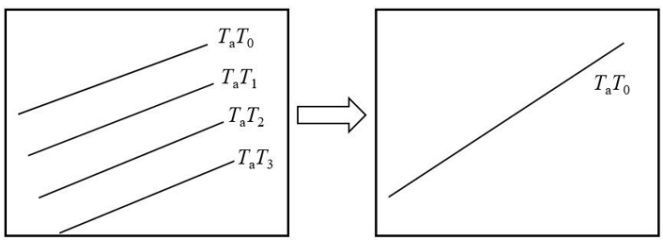


Figure 16. Diagram with the test temperature as the reference

After that, Figure 17 was further processed by the principle of time-aging time equivalence. Taking the aging days of 20 as the reference aging time, the generalized curves of the other aging days were translated to the reference curve, producing the generalized curve at aging temperature 100℃ and test temperature 100℃, that is, the generalized curve E'-lg$f$ after aging for 20 days under 100℃ (Figure 18).

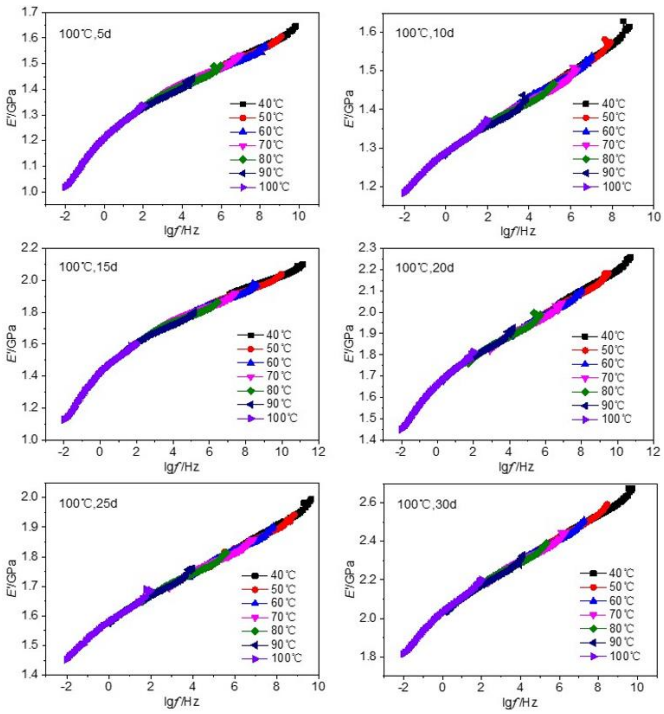


Figure 17. Generalized curves E'-lg$f$ after different aging days at aging temperature of 100℃ (reference temperature: 100℃)

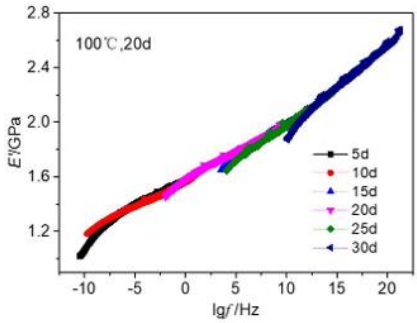


Figure 18. Generalized curves E'-lg$f$ at aging temperature of 100℃ after 20 days

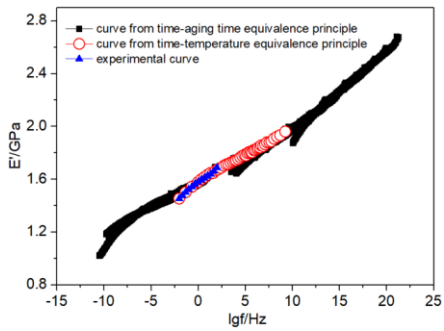


Figure 19. Generalized curve E'-lg$f$ of two accelerated characterizations vs. test curve at 100℃ for 20 aging days

It can be observed that, the change amplitude of test frequency increased from the 4 orders of magnitude (- 2 to 2) in the initial test to 30 orders of magnitude, suggesting that the test time is effectively shortened. The results of the two accelerated characterizations agree well with the test results on the rheological mechanical behavior of our adhesive (Figure 19).

5. CONCLUSIONS

This paper tests the dynamic mechanical properties of the proposed building structure adhesive after thermal aging, and carries out the accelerated characterization of its long-term mechanical properties. The results of the thermal aging test show that, the adhesive properties changed little after aging at 80℃, 100℃ and 120℃ for 30 days. However, the adhesive properties were unstable at 140℃, and declined rapidly with the extension of aging time, indicating that our epoxy structural adhesive is only suitable for the environment cooler than 120℃. Following the principles of time-temperature equivalence superposition and time-aging time equivalence superposition, the authors drew the generalized curve of the epoxy structural adhesive aged for 20 days at 100℃, and increased the variation range of test frequency from 4 orders of magnitude (- 2 to 2) to 30 orders of magnitude, which effectively shortens the test cycle. This research provides an accelerated characterization strategy for the long-term mechanical properties of other polymers, making it possible to obtain the generalized curve of polymers aging for a specific time at a specific temperature.

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