Study on the Testing Method of Relaxation Modulus under Spherical Indenter Loading

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Abstract

Viscoelastic materials are widely used in various fields. In order to better use viscoelastic materials in various working conditions, it is necessary to master the material properties of viscoelastic materials. Relaxation modulus is a characterization method of viscoelastic material properties. In this paper, by analyzing that viscoelastic materials are time-domain related parameters, the load-time curves are obtained by using the spherical indentation instrument to carry out the ramp loading indentation test at different speeds. According to the law of ramp strain history loading, the data at different velocities are summarized and a method for judging the linear viscoelastic boundary of viscoelastic materials in spherical indentation tests is obtained. Aggregate the load-time data at different speeds to obtain a set of valid data for a longer period of time. A method of measuring the relaxation modulus using the ramp-constant strain history is proposed. Compared with the ramp loading test method, it is simple and easy to operate, and it can make up for the short time period of the measured data in the ramp loading test. The experiment verifies the feasibility of the method of ramp-constant strain history.

1. Introduction

Some materials have both viscosity and elasticity, such as rubber and polymers, which are called viscoelastic materials. This characteristic of both viscosity and elasticity is called viscoelasticity, which is the inherent property of materials. In fact, any material has viscoelastic properties, but some are prominently characterized as viscosity, such as lubricants, adhesives, and various fluids, and some are prominently characterized as elasticity, such as spring steel, cast iron, and various metal materials [1, 2].

The viscoelastic properties of materials can be characterized by two parameters: relaxation modulus and creep compliance. Creep compliance can be expressed as the strain response under unit stress. The phenomenon of slow plastic deformation of materials under constant temperature and constant stress for a long time is called creep. In the case of linear materials only, the relaxation modulus means that the stress and strain are proportional at any time during the relaxation process, and the ratio of stress to strain is the relaxation modulus.

Because viscoelastic materials have strong time, temperature, and frequency effects, theoretical analysis, and quantitative calculation are very difficult. Generally, relaxation modulus and creep compliance must be obtained through experiments and data fitting. There is a certain conversion relationship between creep compliance and relaxation modulus, but there is an ill-posed problem in converting creep compliance into relaxation modulus. Small errors in creep compliance will cause large errors in relaxation modulus. Therefore, it is of great significance to directly measure the relaxation modulus of viscoelastic materials.

When solving the relaxation modulus of viscoelastic materials, under the condition that the contact boundary is determined, it can be solved by transforming the viscoelastic problem into an elastic problem. However, considering the change of the contact boundary of viscoelasticity, Lee and Radok [3] used the Hertz contact in the elastic problem to derive the viscoelastic parameter solution when the rigid spherical indenter is pressed into a linear viscoelastic, isotropic uniform half space, and the area of the contact area...
does not decrease. Hunter [4] derived the viscoelastic solution under the condition that the contact area is from maximum to minimum when the rigid spherical indenter is retracted. Ting [5] derived the viscoelastic solution of a rigid axisymmetric indenter pressed into a linear viscoelastic half-space at any time on the basis of both of them. The development of these theories also laid the foundation for the indentation instrument to measure the relaxation modulus of viscoelastic materials.

The ideal relaxation modulus measurement is to apply a step strain \( \varepsilon = \varepsilon_0 \) to the material under certain conditions and then calculate the time-dependent stress \( \sigma(t) \). When the measured material is a linear viscoelastic material, the relationship between stress and strain is as follows:

\[
\sigma(t) = \varepsilon_0 E(t).
\]

The function \( E(t) \) is called relaxation modulus, which represents the stress required to produce and maintain unit strain. It is usually a monotonic decreasing function, or at least a nonincreasing function related to time. As shown in (1), in order to obtain the relaxation modulus of the material, a certain strain must be applied to the material at zero time. But in reality, this condition is impossible. We can only ensure that the strain is applied in as little time as possible to realize the measurement of load. This is also an important problem in the measurement of relaxation modulus by indentation instruments.

When the indentation experiment is performed, the rigid spherical indenter completes the deformation from the beginning of the indentation to the maximum indentation position and must experience the deformation time \( t_0 \). A large number of scholars have studied and analyzed this, hoping to reduce the influence of the strain application process on the relaxation modulus test process through a reasonable method.

Some scholars used the step strain history method of the indentation instrument to measure the relaxation modulus by correcting the data in the relaxation stage [6–10]. The correction method of Zapas and Phillips [6] is the simplest, which is calculated by moving the relaxation area forward for a certain time range. Of course, some scholars calculate the relaxation modulus by adding correction factors [8–10]. Among them, the best method is to calculate the relaxation modulus by using the measured data with the minimum error between the relaxation data after \( 5t_0 - 10t_0 \) and the ideal step loading in a recursive manner [6, 7].

Zhao [11] verified through experiments that the error between the data after the ramp loading history and the ideal step experimental data are less than 1% after \( 5t_0 - 10t_0 \). Smith [12, 13] showed that the relaxation modulus of a linear viscoelastic material could be related to the slope of the stress–strain curve obtained from a simple uniaxial tensile test. Shahani et al. [7] mentioned that the ramp loading history in the stress relaxation test is similar to the loading conditions in the uniaxial tensile test, so the relaxation modulus function can be obtained through the stress relaxation data in the ramp history loading region. Huang and Lu [14] measured the relaxation modulus of viscoelastic materials by using the data measured in the ramp strain history method.

At present, the relaxation modulus of spherical indentation instruments is mainly measured by the step strain history method and the ramp strain history method. The ramp loading method is limited by the linear viscoelastic interval and the measurement time is limited. The relaxation test under constant velocity loading needs to be corrected by the correction factor, and the range of the measured relaxation modulus is limited, which means that it cannot reach the full time domain of the relaxation modulus measurement. In the measurement process, the data validity of the ramp loading part are also ignored. Therefore, some scholars have proposed combining the two methods to measure the relaxation modulus of viscoelastic materials by using the method of ramp-constant strain history [15, 16].

When judging the linear viscoelasticity interval of viscoelastic materials, Chen [17] and Zhu [18] took the sudden change of storage modulus during the DMA (dynamic mechanical analysis) measurement as the critical point of the linear viscoelasticity interval. Lu et al. [19] used an electron microscope to observe the recovery process of the indentation during the measurement process and regarded the fully recovered indentation as the measurement in the linear viscoelasticity range.

For the linear viscoelastic interval measurement of viscoelastic materials in the two measurement methods, the DMA test is expensive and complicated to measure the linear viscoelastic boundary of materials. It measures the overall deformation of the sample, which is obviously different from the deformation in the indentation test. On the other hand, by observing whether the indentation is completely recovered, human factors have a great influence.

Therefore, the load-time data of the spherical indentation instrument at different speeds are compared by using the method described in this paper, and it is found that the linear viscoelastic formula is satisfied only when the indenter displacement of the rigid spherical indenter is constant so as to judge the boundary of the linear viscoelastic. Combining with the ramp strain history methods and step strain history methods, a set of formulas for solving the relaxation modulus through the ramp-constant strain history are proposed, and the feasibility of the formula is verified by the experimental comparison with the ramp strain history.

1.1. Theoretical Source of Test Formula. Hertz contact theory is proposed by Hertz in the study of the relationship between strain and stress in the contact part of two glass lenses in contact under the action of force [20]. By extending the Hertz contact theory to the test of viscoelastic materials with rigid spherical indenter and plane viscoelastic materials, it can be concluded that the load-displacement relationship is as follows:

\[
P = \frac{4\sqrt{R}}{3(1 - \nu^2)}Eh^{3/2}.
\]
where $R$ is the radius of the spherical indenter, $\nu$ is the Poisson’s ratio ($\nu = 0.5$), $h$ is the indentation depth, $E$ is the elastic modulus of materials, and $P$ is the load.

According to the research of Lee and Radok [3], we can get that the load-displacement relation for spherical indentation is as follows:

$$P(t) = \frac{4\sqrt{R}}{3(1 - \nu^2)} \int_0^t E(t - \xi) \frac{dh^{3/2}}{d\xi} d\xi. \quad (3)$$

The pressing law of rigid spherical indenter can be expressed by step function $H(t)$, when $t > 0$ ($t < 0$), $H(t) = 1(0)$. Therefore, the actual loading history of the rigid spherical indenter can be expressed as follows:

$$P(t) = V_0 t H(t) - V_0 (t - t_0) H(t - t_0), \quad (4)$$

when $0 < t < t_0$, by substituting (4) into (3), the load-displacement relation can be obtained.

$$P(t) = \frac{2V_0^{3/2} \sqrt{R}}{1 - \nu^2} \int_0^t E(\xi) \sqrt{1 - \xi^2} d\xi. \quad (5)$$

Relaxation modulus can be expressed according to the generalized Maxwell model as follows:

$$E(t) = E_{\infty} + \sum_{i=1}^{N} E_i e^{-\lambda_i t}. \quad (6)$$

Substitute (6) into (5), the formula of ramp loading part can be expressed as follows:

$$P(t) = \frac{2V_0^{3/2} \sqrt{R}}{1 - \nu^2} \left( \frac{2}{3} E_{\infty} t^{3/2} + \sum_{i=1}^{N} E_i e^{-\lambda_i t} \sqrt{1 - \xi^2} \right). \quad (7)$$

When $t > t_0$, the rigid spherical indenter remains constant at its maximum displacement, the displacement is as follows:

$$h(t) = V_0 t - V_0 (t - t_0) = V_0 t_0. \quad (8)$$

As shown in Figure 1, Lee and Knauss [21] used the ramp-constant strain history to divide it into the combination of two ramp strain histories and deduced a method using reverse recursion to obtain the relaxation modulus in the whole-time domain. The ramp-constant strain process can be divided into the difference between two ramp strain histories. Du et al. [22] divided $h(t)$ into two parts in the relaxation stage when $t \geq t_0$ by using the method of strain division.

$$P(t) = \frac{2R}{1 - \nu^2} \int_0^t E(t - \xi) \frac{dh(t)}{d\xi} d\xi - \frac{2R}{1 - \nu^2} \int_{t-t_0}^t E(t - \xi) \frac{dh(t)}{d\xi} d\xi. \quad (9)$$

By introducing (6) into (9), Du et al. obtained the indentation law of the flat punch in the relaxation stage and obtained the equation.

$$P(t) = \frac{2Rv_0}{1 - \nu^2} \left[ E_{\infty} t_0 + \sum_{i=1}^{N} E_i \left( e^{-\lambda_i (t-t_0)} - e^{-\lambda_i t} \right) \right] (t \geq t_0). \quad (10)$$

Mattice et al. [9] used the step correction coefficient existing in the creep derivation process to introduce it into the relaxation modulus step loading method under the spherical indentation and obtained the formula of step loading (the ramp loading time is not higher than 5% of the relaxation time) by analogy. Therefore, by analogy with the methods of Du et al. [22], we calculate (5) recursively to obtain the following formula:

$$P(t) = \frac{2RV_{0t}^{3/2}}{1 - \nu^2} \int_0^t E(\xi) \sqrt{t - \xi} d\xi - \frac{2RV_{0t}^{3/2}}{1 - \nu^2} \int_{t-t_0}^t E(\xi) \sqrt{t - \xi} d\xi. \quad (11)$$

Substitute (6) into (11) to obtain the equation of relaxation stage in the loading history of rigid spherical head.

$$P(t) = \frac{2RV_{0t}^{3/2}}{1 - \nu^2} \left( \frac{2}{3} E_{\infty} t_0^{3/2} + \sum_{i=1}^{N} E_i e^{-\lambda_i t} \sqrt{t - \xi} \right). \quad (12)$$

Finally, we can obtain the formula from the beginning of ramp loading to the end of relaxation time is as follows:

Ramp loading part:

$$P(t) = \frac{2RV_{0t}^{3/2}}{1 - \nu^2} \left( \frac{2}{3} E_{\infty} t_0^{3/2} + \sum_{i=1}^{N} E_i e^{-\lambda_i t} \sqrt{t - \xi} \right) \left( t < t_0 \right). \quad (13)$$

Relaxation part:

$$P(t) = \frac{2RV_{0t}^{3/2}}{1 - \nu^2} \left( \frac{2}{3} E_{\infty} t_0^{3/2} + \sum_{i=1}^{N} E_i e^{-\lambda_i t} \sqrt{t - \xi} \right) \left( t \geq t_0 \right). \quad (14)$$

2. Experimental Knowledge

2.1. Selection of Delay Function. There are many models of viscoelastic materials. The ramp loading method and ramp-constant loading method in this paper choose the generalized Maxwell model composed of a spring and multiple Maxwell models in parallel to analyze. The material relaxation modulus function equation under the generalized Maxwell model is as follows:

$$E(t) = E_{\infty} + \sum_{i=1}^{N} E_i e^{-t/\tau_i}. \quad (15)$$

where, $E_{\infty}$ is the relaxation modulus parameter when $t$ is infinite, $\tau_i$ is the relaxation time, and $E_i$ is the relaxation modulus parameter of each time period. Among them, the range of $i$ is determined by the time period in the test process and will affect the choice of relaxation time.

Knauss [23] analyzed and introduced in detail that in the process of fitting using MATLAB, the relaxation time will only affect a certain period of time in the relaxation modulus function. It is concluded that choosing a relaxation time in every ten times time interval is sufficient to meet the requirements. The selection of relaxation time will be affected
by the time period of measurement data. After analysis and verification, Knauss believes that it is necessary to expand 100 times at both ends of the time period of the measured data to select the relaxation time $\tau_i$.

2.2. An Expansion Method of Effective Time Domain of Measurement Data. If we want to better grasp the characteristics of viscoelastic materials, we need the material characteristics of materials at any time, which requires us to master the change of relaxation modulus function of viscoelastic materials in a long-time range. This also means we need the indentation instrument to measure the data in a long-time range. The method of measuring relaxation modulus by the indentation method is applicable under the condition of linear viscoelasticity. Therefore, due to the excessive deformation of viscoelastic materials during the indentation experiment, the time period of data obtained from each measurement of ramp loading is fixed and limited. In order to better grasp the material properties of viscoelastic materials, this paper will summarize the data obtained from multiple groups of ramp measurements using the methods mentioned below to expand the effective period of measurement data.

According to (5), we can get the following:

$$\int_0^t E(\xi) \sqrt{1 - \xi^2} d\xi = P(t) \frac{1 - \gamma^2}{2V_0^{3/2} \sqrt{R}}. \quad (16)$$

The relaxation modulus function of viscoelastic material is determined by the inherent properties of the material, which is the same and constant in the same time period. Through the analysis in Section 2.1, the relaxation modulus parameter and relaxation time are valid in a certain period of time. Therefore, in the same time period, the right side of the above (16) is the same at different speeds. Write the right formula as follows:

$$K = P(t) \frac{1 - \gamma^2}{2V_0^{3/2} \sqrt{R}}. \quad (17)$$

The above law can be used to judge the rough displacement of indenter in the indentation test of viscoelastic materials, and the test data at different speeds can be summarized according to the time law so as to obtain longer time effective experimental data.

3. Experimental Materials and Settings

3.1. Selection of Experimental Materials. The viscoelastic material used in indentation test is vulcanized rubber block,
with Shore hardness of 74HA, tensile strength of 1.5 MPa, and size of $100 \times 100 \times 10$ mm. As shown in Figure 2, the surface of the pressed material is smooth (no concave convex can be observed by naked eye).

3.2. Experimental Setting. The device shown in Figure 3 is used for the experiment. The maximum pressing displacement of the spherical indenter can reach 1800 microns, the maximum load that can be measured is not less than 50 N, the force at the millinewton level can be measured, and the speed range can be controlled to about $0.1-1000 \mu m/s$. The radius of the rigid ball used in the experiment is 4 mm.

All the experiments were carried out at room temperature (26°C), and due to various errors that may exist in the experimental process and the impact of the environment, the experiments were carried out many times, and some groups of data were selected from them to use after averaging. The specific experimental process can be divided into five parts:

1. Several experiments were carried out with an indentation instrument, and the load-time curve was compared to determine the repeatability of the experimental device during the experiment.

2. Large displacement indentation experiments at three different speeds were carried out on the material, and the compression displacements conforming to linear viscoelastic conditions were judged by the judgment method in 2.2.

3. The indentation test is carried out in the achievable speed range by using the experimental device, and multiple sets of data are obtained to expand the shortage of short measurement time of single set of data in ramp loading. Multiple sets of experimental data are summarized into a long-time range of effective data.

4. The ramp-constant experiment was carried out within the restricted compression displacement, and the loading history of the ramp-constant experiment is shown in Figure 1(a). The experimental data were fitted by the least square method in Matlab and the proposed formula of ramp-constant strain history and the relaxation modulus function of rubber material was obtained.

5. By using the relaxation modulus function of rubber material to fit the data of ramp loading history measurement, the correctness of the proposed formula is verified.

4. Analysis of Experimental Results

4.1. Judgment and Analysis of Repeatability. The relaxation modulus test device as shown in Figure 4 is used to make the rigid spherical indenter perform the indentation experiment with 1 mm displacement at the speed of $100 \mu m/s$. Multiple sets of experimental data were measured and then compared and analyzed. As can be seen from the measurement results shown in Figure 4, the relative changes among the experimental results are small, and the experimental data have repeatability, which can be used for subsequent experiments.

In the process of ramp loading measurement, the measured load-time data will be affected by the experimental device, the minimum reading time will be affected by the sensor sensitivity, and the maximum reading time will be limited by the sensor range.

4.2. Judgment of Pressing Displacement. When the rigid spherical indenter is pressed into the material within a certain displacement range, the rubber viscoelastic material exhibits linear viscoelasticity. In this experiment, the pressed displacement was roughly judged by the method mentioned in Section 2.2. By measuring load-time curves of ramp strain history at different speeds $10 \mu m/s$, $100 \mu m/s$, and $1000 \mu m/s$, a comparative analysis of $K$ value was conducted according to the extended effective time domain method in Section 2.2. The experiment was carried out on a simple device designed and installed by ourselves. According to the comparison, the approximate pressing displacement can be obtained, and the pressing displacement can be appropriately corrected in the subsequent experiments for the ramp-constant strain experiment.

As shown in Figure 5 and Table 1, it can be seen that the $K$ value time curve under the ramp strain history with the speed of $10 \mu m/s$ and $100 \mu m/s$ increases with time, and the difference between the two $K$ values becomes larger and larger. According to Table 1, the error of 10 and 100 changes significantly after 1.2s.

As shown in Figure 6 and Table 2, there is the same regular change as in Figure 5 under the ramp strain history of $100 \mu m/s$ and $1000 \mu m/s$. According to Table 2, at 0.2 s, the relative error between $K$ values obtained at $100 \mu m/s$ and $1000 \mu m/s$ speeds exceed 20%.

By analyzing the above measurement results, it can be judged that when the indentation displacement is $100 \mu m-200 \mu m$, the indentation displacement boundary of the material from linear viscoelastic material to nonlinear viscoelastic material can be obtained from three different speed changes.

4.3. Integrate Data of Ramp Strain History. According to the analysis, it can be concluded that the linear viscoelastic boundary of the rubber material used this time is about
100 \mu m–200 \mu m, with 200 \mu m as the maximum rigid ball head pressing displacement, every 10 times the speed between 0.1 \mu m/s and 1000 \mu m/s measure a set of data. After that, different measurements at different speeds in the linear viscoelastic interval can be combined into a long-time effective data to solve the viscoelastic relaxation function of rubber materials.

According to the load-time data with loading speeds of 0.1 \mu m/s, 1 \mu m/s, 10 \mu m/s, 100 \mu m/s, and 1000 \mu m/s, the \( K \) value data were solved, and the \( K \) values at different time periods were summarized to obtain the \( K \) value function curve as shown in Figure 7. From these data points, the valid \( K \) value data for a long time period can be summarized.

4.4. Analysis of Ramp-Constant Strain History. As shown in Figure 8, the ramp-hold strain history experiment was conducted within the range of linear impingement displacement. Historical loading of ramp strain was carried out at the speed of 100.38 \mu m/s, and the maximum displacement was reached at 0.86 s. After that, the strain was kept unchanged for relaxation part.
experiment, and a set of ramp-constant data of 0.02s–1600s was obtained.

When fitting the data in the period of 0.02s–1600s, we need to determine the specific value of the relaxation time in (13 and 14). According to Section 2.1, take a relaxation time every ten times the time range and expand both ends of the measured data time period by 100 times as the range of relaxation time, where the relaxation time $\tau$ take $10^{-4}$, $10^{-3}$, $10^{-2}$, $10^{-1}$, $10^0$, $10^1$, $10^2$, $10^3$, and $10^4$.

In order to prevent the influence of load size on fitting effect, the optimization condition of MATLAB fitting program is set as relative error. In this way, the influence of load size on the optimization conditions of fitting can be reduced, and the uneven error in each time period caused by different load force size can be avoided.

As shown in Figures 9 and 10, the relative error of the fitting result is less than 4%. According to the fitting, the relaxation modulus function of viscoelastic material is as follows:

$$E(t) = 2.0982 + 1.0651e^{-10t} + 2.4093e^{-t}$$

$$+ 0.8614e^{-0.1t} + 0.9156e^{-0.01t} + 0.5122e^{-0.001t}$$

$$+ 1.1848e^{-0.0001t} + 2.4834e^{-0.00001t}.$$
Table 2: Detailed comparison of 100 μm/s data and 1000 μm/s data.

<table>
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<tr>
<th>Time/t</th>
<th>( K ) value at 100 μm/s</th>
<th>( K ) value at 1000 μm/s</th>
<th>Relative error (( (A - B)/B \times 100% ))</th>
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<td>0.02</td>
<td>17410.92</td>
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</tr>
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<td>170811.5</td>
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<td>224635.7</td>
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<td>535817.4</td>
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Figure 7: Data segment integration at different speeds.

Figure 8: Ramp-constant strain history.

Figure 9: Data fitting diagram of ramp-constant strain history.

Figure 10: Relative error of data fitting under ramp-constant strain history.
In order to verify the feasibility of the proposed ramp-constant strain history formula, the relaxation modulus function obtained by this method is used to predict the $K$ value data of the ramp strain history from 0.02s to 1600s and compare it with the measured value. As shown in Figure 11 and 12, the relaxation modulus function $E(t)$ obtained by using the ramp-constant strain history formula has a good fitting effect on the effective data of $K$ value integrated with ramp loading history. Therefore, the ramp-constant strain history formula proposed in this paper has the same validity as the ramp strain history formula.

Compared with the ramp strain history method, the ramp-constant strain history method is simpler and more convenient in the number of experiments. During the ramp strain experiment, the time of single experiment data is affected by the size of the pressing displacement, while the ramp-constant strain can be measured for a long time after the maximum displacement is reached, without the limitation of the maximum measurement time.

5. Conclusions

In this paper, the measurement method of the relaxation modulus of viscoelastic materials is studied. That is, the measurement process and feasibility of the method of solving the relaxation modulus by the ramp-constant strain using a spherical indenter. In the measurement process, the measurement of the linear viscoelastic boundary of viscoelastic materials is extremely important. In this paper, through the derivation and analysis of the theoretical formula, it is found that there is a certain conversion relationship between the load-time data measured at three different speeds of 10 $\mu$m/s, 100 $\mu$m/s, and 1000 $\mu$m/s so that the linear viscoelastic boundary of viscoelastic materials can be judged. The implementation of the ramp-constant strain method is ensured by the definition of the linear viscoelastic boundary, and tests were carried out with vulcanized rubber. The ramp-constant strain history method and the ramp strain history method are compared. It is found by comparison that the proposed ramp-constant strain history method and the ramp strain history method have measurement errors within 10% and have the same ability to quantify the relaxation modulus of viscoelastic materials as the ramp strain history method. However, compared with the ramp strain history method, the proposed method measures the relaxation modulus of materials in a longer time domain and is simpler.

6. Discussion

Possible limitations of the ramp-constant strain history method are as follows:

(1) The selection of the speed when determining the linear boundary. It is necessary to measure the linear boundary by comparing the overlapping parts
between different speeds, the size relationship between adjacent speeds cannot be too small (the difference between the results is not obvious) or too large (the overlapping parts are too small, which is difficult to judge). In actual measurement, the relationship between adjacent speeds is generally better at 10 times.

(2) The size of the specimen. In the indentation test, in order to ignore the influence of the material boundary conditions when the material is compressed, $a/d > 10$ should be made (a is the side length of the square sample and $d$ is the diameter of the rigid spherical indenter).

(3) The length of relaxation time in the slope constant strain history method. Through the analysis of the experimental results in this paper, the fitting error near $t_0$ is the largest. In order to minimize the influence of this part on the relaxation modulus measurement results. In actual measurement, the time of relaxation area should be greater than $5t_0$, and the longer the time is, the better the effect will be.

**Data Availability**

The datasets used and analyzed during the current study are available from the corresponding author upon reasonable request.

**Conflicts of Interest**

The authors declare that they have no conflicts of interest.

**References**


