Research on Measurement Traceability Method of Dynamic Shear Rheometer

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Abstract. The dynamic shear rheometer is the basic instrument for studying viscoelastic materials. In this paper, the influences of the accuracy of dynamic shear rheometer were analyzed, calibration parameters were proposed and verified through experiments, and the extended uncertainty was 3.0 (k=2), which verified the feasibility of using viscosity reference materials as a technology to realize the measurement traceability of dynamic shear rheometer, and the traceability method can be promoted and applied to the traceability of various equipment.

Keywords: metrology, dynamic shear rheometer, measurement traceability, complex shear viscosity, calibration method; uncertainty.

1. Introduction

With the continuous improvement of China's transportation network, asphalt pavement has become a common form in modern highway construction due to its advantages such as seamless, comfortable driving, wear resistance, easy maintenance, and renewable utilization. Asphalt and asphalt mixture is a thermorheologically simple material, the cracking, rutting, fatigue and other damage problems of asphalt pavement are all related to the viscoelastic properties of asphalt mixture. Therefore, the viscoelastic property of asphalt mixtures is particularly important [1] [2].

In the specifications of asphalt binder road performance from the US Strategic Highway Research Program (SHRP), DSR (dynamic shear rheometer) is used for the first time to evaluate the high-temperature performance and low-temperature fatigue performance of asphalt binders, and then carried out the performance grade [3] [4], the PG system has a good correlation with the pavement performance of asphalt, and it has been applied in most of the highway construction in china [5] [6].

DSR is a basic instrument for studying viscoelastic materials, which is suitable for the measurement of rheological parameters such as asphalt complex shear viscosity. The SHRP PG system standard of asphalt and JTG E20-2011 “Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering” and JTG F40-2004 “Technical Specification for Construction of Highway Asphalt Pavements”, are mainly used to evaluate the high-temperature performance before and after the aging of film oven and the fatigue performance (medium temperature performance) of asphalt after the aging of pressure [7-9]. In foreign countries, the DSR calibration/testing is mainly according to the standards of AASHTO T315, AASHTO T350, AASHTO TP101, ASTM D7175, ASTM D7405, DIN EN 14770, AASHTO T316, ASTM D4402 and DIN EN 13302. As a widely used testing equipment, however, there are no national standards and metrology technical regulations of DSR in China. Therefore,
it is impossible to evaluate the equipment situation in the market, take the imported equipment according to AASHTOT 315 used for external sharing operation in a university for example, which use the CANNON N2700000SP as the viscosity reference materials, and the relative indication error of the test is between 9.64% and 19.95%, which is far higher than the specification requirements. At present, there is no measurement method of DSR in China, so the equipment cannot be traced effectively. This paper introduces the dynamic shear rheometer, including the analysis of the influences of accuracy, traceability path, calibration method, and uncertainty evaluation.

2. Working principle
At present, the dynamic shear rheometers used in my country are mainly produced by Malvern Panalytical Company, Anton Paar Company, TA INSTRUMENTS, British Bohlin Company, German Thermo Fisher Company, Tianjin Gangyuan Test Instrument Factory and other companies. The dynamic shear rheometer is composed of control and data acquisition system, rotor, test board, loading device, temperature control system, etc. The working principle of the dynamic shear rheometer is that: a sinusoidal alternating stress is applied to the asphalt sample, a sinusoidal alternating strain force is generated, and there is a phase difference between the two stresses, then the relevant parameters of the dynamic rheological performance of the asphalt are measured. The bitumen sample is sandwiched between the oscillating rotor and the test plate when the equipment is working, and the motion trajectory of the rotor is cycling from point A to point B to point A to point C, the working principle of the dynamic shear rheometer is shown in Fig. 1.

![Figure 1. Schematic Diagram of the Working Principle of the Dynamic Shear Rheometer](image_url)

The results of the dynamic shear experiment can be expressed as the complex shear modulus according to (1).

\[ G^* = G' + iG'' \]  

(1)

Where the \( G' = G'\cos\delta \) is the storage shear modulus, \( G'' = G'\sin\delta \) is the loss shear modulus which expresses the energy loss of asphalt during the deformation process of asphalt, \( \delta \) is the phase angle, and the larger the value of \( G'\sin\delta \), the faster the energy loss under repeated loads.

3. Calibration parameters
3.1. Influence of rotor size
In the asphalt test, there is a gap of 1mm between the rotor and the metal plate. The size of the rotor directly affects the measurement accuracy of DSR, and indirectly affects the evaluation results of the asphalt sample. The rotor is made of stainless steel or aluminum alloy, the surface is smooth and polished, and the nominal diameters are 8.00mm and 25.00mm respectively, and the rotor and the test plate should be concentric, and there should be no horizontal and vertical swings when rotating.
JTG E20-2011 “Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering” requires two specifications of metal plates with smooth surfaces for test panels, one piece has a diameter of 8.00mm±0.05mm, and the other one has a diameter of 25mm±0.05mm. In addition, AASHTO T315 points out that for a rotor with the nominal diameter of 25mm, a diameter error of ±0.05mm will result in a 0.8% complex modulus error, and a rotor with a nominal diameter of 8mm with the diameter error of ±0.01, ±0.02, and ±0.05mm will result in complex modulus errors of 0.5%, 1.0%, and 2.5% respectively, which are shown in the Fig. 2. Therefore, the diameter of the rotor is generally 8.00mm±0.05mm and 25mm±0.05mm, since the rotor will not be deformed under normal circumstances, the rotor size requirements should be specified in the product standard combined with expert opinions.

3.2. Influence of Temperature
Temperature has a strong influence on asphalt [10]. Cold asphalt looks like an elastic material, and high temperature asphalt looks like a viscous material. When asphalt is heated to 200°C, the viscosity of the asphalt is as less as $10^{-1}$Ps·s. However, the asphalt in the severe cold state is close to solid, and the viscosity is as high as $10^{11}$ Ps·s. Therefore, DSR must precisely control the temperature during the measurement.

![Figure 2. Influence of Gap Error](image)

The temperature control system of DSR is used to control the temperature change in the detection process by controlling heating (stage or progressive) or cooling (stage or progressive) to maintain a constant temperature environment. The temperature control system generally uses circulating liquid or adjustable gas to control the temperature. The technical requirements of JTG E20-2011 for DSR temperature control are as follows: a) Environment room: The environment room is used to control the temperature of the sample during the test, and maintain a constant sample environment through heating or cooling. The heating or cooling medium in the environmental chamber should be liquid or gas that does not affect the properties of asphalt. b) Temperature controller: the temperature of the sample should be controlled within ±0.1°C of the test temperature in the temperature range of 5°C~85°C. C) Temperature sensor: the indication error of sensor is ±0.1°C. In addition, AASHTO T315 indicates that the indication error of temperature is ±0.1°C. Therefore, the temperature indication error of DSR is ±0.1°C.

3.3. Influence of loading process
In general, the loading method of DSR is to apply a sinusoidal oscillation load to the sample at (10±0.1 rad/s) frequency, which should be accurate to 1% if other frequencies are used. And the loading process
should provide a stress controlled load or a strain controlled load. If it is a strain-controlled load, a periodic torque should be provided during the loading process, and the angular rotation strain accuracy should be less than 100μrad. If it is a stress-controlled load, the loading process shall provide a cyclic torque with an accuracy not greater than 10mN.m. The stress has a great influence on the test result during the loading process, since the loading equipment is generally packaged in the DSR case, it is not economical and universal to disassemble the loading equipment for calibration. Therefore, indirect calibration should be considered. According to JTG E20-2011 “Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering” the loading equipment of DSR is required to apply sinusoidal vibration load of 10rad/s±0.1rad/s to the sample, and the loading method can be a stress controlled load or a strain controlled load.

In summary, the calibration parameters of the equipment are as follows: the indication error of temperature is ±0.1℃, the relative indication error of complex shear viscosity is ±3%, and the repeatability of complex shear viscosity is no more than 3%.

4. Measurement traceability

Based on the analysis of influences of the equipment accuracy, the following technical requirements of the standard device should be adopted: the maximum allowable error of thermometer is ±0.05℃, and the nominal value of dynamic viscosity ranges from 50000 mPa·s to 300000 mPa·s when the constant temperature of the viscosity reference material is 64℃, the block diagram of the measurement traceability is shown in Fig.3.

5. The process and results of Calibration

Make sure that the calibration ambient temperature is 23℃±5℃, and the ambient humidity is no more than 85% RH. The dynamic shear rheometer of a certain manufacturer is taken as an example. The appropriate viscosity reference materials was transferred to the test plate, then the measurement temperature of DSR was set at 64℃, the angular frequency was set at 10 rad/s, and the amplitude was set at 12% in principle. The value of DSR complex shear viscosity $\eta_i^*$ was measured and recorded.

Repeat the test according to the above steps for three times, then the average $\eta^*$ was calculated as the measurement result of complex shear viscosity, the test data are shown in Table 1. The relative indication error $\delta$ of the complex shear viscosity was calculated according to (2), and the relative indication error $\delta_i$ of the $i$-th measurement was calculated according to (3) and (4).

$$\delta = \frac{(\eta^*-10\eta)}{10\eta} \times 100\%$$

Where the $\delta$ is the relative indication error of complex shear viscosity, $\eta$ is the motion viscosity value of a standard substance, mPa·s.
\[ s = \frac{\delta_{i_{\text{max}}} - \delta_{i_{\text{min}}}}{c\sqrt{n}} \]  
\[ \delta_i = \frac{(\eta_i^0 - \eta_i^1)}{10\eta} \times 100\% \]

Where \( s \) is the standard deviation of repeatability. \( \delta_{i_{\text{max}}} \) is the maximum value of the relative indication error of the complex shear viscosity in the three measurements, \( i = 1, 2, 3 \). \( \delta_{i_{\text{min}}} \) is the minimum value of the relative indication error of the complex shear viscosity in the three measurements, \( i = 1, 2, 3 \). \( C \) is the range coefficient, take 1.69. \( n \) is the measurement time, take 3. \( \delta_i \) is the relative indicating error of the complex shear viscosity of the \( i \)-th measurement.

It can be found from the table 1 that the relative error of the complex shear modulus of the calibrated dynamic shear rheometer is less than ±3%. The calibration results meet the corresponding technical requirements.

| NO. | Measuring Temperature (℃) | Motion viscosity value of standard substance (mPa·s) | A Factory Dynamic shear rheometer measurements (mPa·s) | Relative indication error of complex shear viscosity |
|-----|--------------------------|-----------------------------------------------------|---------------------------------------------------|--------------------------------------------------|
| 1   | 52                       | 649800                                              | 6512689.46                                         | 0.84%                                            |
|     |                          |                                                     | 6512689.34                                         |                                                  |
|     |                          |                                                     | 6512697.75                                         |                                                  |
| 2   | 58                       | 427600                                              | 4295277.35                                         | 0.45%                                            |
|     |                          |                                                     | 4295287.64                                         |                                                  |
|     |                          |                                                     | 4295284.35                                         |                                                  |
| 3   | 64                       | 289600                                              | 2855881.21                                         | -1.39%                                           |
|     |                          |                                                     | 2855880.47                                         |                                                  |
|     |                          |                                                     | 2855887.17                                         |                                                  |
| 4   | 70                       | 199200                                              | 1990697.46                                         | -0.07%                                           |
|     |                          |                                                     | 1990699.65                                         |                                                  |
|     |                          |                                                     | 1990697.26                                         |                                                  |
| 5   | 76                       | 139000                                              | 1406840.55                                         | 1.21%                                            |
|     |                          |                                                     | 1406853.05                                         |                                                  |
|     |                          |                                                     | 1406842.55                                         |                                                  |

6. Evaluation of measurement uncertainty

The uncertainty sources of the dynamic shear modulus measurement indication include the uncertainty \( u_{r1} \) of viscosity reference material and the uncertainty \( u_{r2} \) of measurement repeatability. The measurement model is shown in (2).

6.1. Uncertainty evaluation

(1) Uncertainty \( u_{r1} \) of viscosity reference material

Assuming that the measurement uncertainty is \( u_{r1} = m\% \), then the standard uncertainty of the viscosity reference materials is \( u_{r1} = \frac{u_{r1}}{k_1} \), according to the type B evaluation of uncertainty, \( k_1 = 2 \).

(2) Uncertainty \( u_{r2} \) of measurement repeatability

The uncertainty of measurement repeatability is evaluated by the type an evaluation method. The viscosity standard substance is selected to carry out the repeatability test, and the standard deviation is calculated according to the range method, the uncertainty of measurement repeatability is calculated according to the (5).
\[ u_{r2} = \frac{s}{\eta} \times 100\% \] (5)

Take the data in table 2 for example, we can get the \( u_{r2} = 0.20\% \).

**Table 2.** test data of Measurement repeatability

| NO. | Value (mPa·s) | Measurement repeatability s (mPa·s) | \( u_{r2} \) |
|-----|--------------|-------------------------------------|--------------|
| 1   | 291400.0     | 277700.0                            | 275800.0     |
|     | 9512.195     | 0.20%                               |              |

**Table 3.** Summary table of uncertainty components of DSR calibration results

| NO. | uncertainty sources                  | Uncertainty component (%) | Type | distribution |
|-----|--------------------------------------|---------------------------|------|--------------|
| 1   | uncertainty of viscosity reference material | \( u_{r1} = \frac{U_{r1}}{k_1} \) | B    | normal       |
| 2   | uncertainty of measurement repeatability | \( u_{r2} = \frac{s}{\eta} \times 100\% \) | A    | /            |

(3) Summary of measurement uncertainty components
The uncertainty components of the DSR calibration results are summarized in Table 3.

6.2. Combined standard uncertainty \( u_{rc} \)
The combined standard uncertainty is shown in (6).

\[ u_{rc} = \sqrt{u_{r1}^2 + u_{r2}^2} \] (6)

6.3. Expanded uncertainty
The expanded uncertainty is shown in (7), take the \( k=2 \),

\[ U_{rel} = k \cdot u_{rc} \] (7)

7. Conclusion
In this paper, the measurement traceability of DSR was conducted, the calibration parameters were proposed and verified through analyzing the influences of the accuracy of the dynamic shear rheometer, and the extended uncertainty was 3.0 (\( k=2 \)), which verified the feasibility of using viscosity reference materials as a technology to realize the measurement traceability of the dynamic shear rheometer, and the traceability method can be promoted and applied to the traceability of various dynamic shear rheometers.

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