Comparison of Different DSR Protocols to Characterise Asphalt Binders

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Abstract The use of Dynamic Shear Rheometer (DSR) has become a standard tool to characterise bituminous binders, either for research or specifications purposes. Testing with the DSR enables to cover a wide range of temperature and frequency conditions in a fast way by using limited amount of materials. Depending of the test type, DSR measurements provide different rheological parameters to characterise physically the materials either at low, intermediate or high temperatures. At the same time, different test protocols have emerged. While the preferred mode is running in frequency sweep at different temperatures, the test can also be run with a temperature ramping at a fixed frequency. In order to evaluate the validity of the DSR testing mode, four different test protocols were run, including a frequency sweep test at different temperatures and temperature ramping at a fixed frequency with different temperature rates. This experiment was applied to three different bituminous binders, two non-modified binders, soft and hard, and a standard Polymer modified Bitumen (PmB). The analysis was conducted on shear modulus and phase angle versus Temperature. The outcomes showed a reasonably good correlation between the different protocols in the common range of temperatures and demonstrated instrument limitations regarding geometry and control parameters.

Keywords Bitumen · Polymer modified bitumen · DSR · Rheology

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H. Di Benedetto et al. (eds.), *Proceedings of the RILEM International Symposium on Bituminous Materials*, RILEM Bookseries 27,
https://doi.org/10.1007/978-3-030-46455-4_58

1 Introduction

For a long time, only empirical testing, such as penetration value, softening point temperature, ductility and Fraass breaking point temperature, have been used for bituminous binder characterisation. As part of the SHRP-program (Strategic Highway Research Program) [1], new rheological test methods where introduced for bituminous binder testing. Especially the Dynamic Shear Rheometer (DSR) has become a standard test instrument to assess material properties based on fundamental rheological behaviour. The DSR is a very versatile instrument, which covers a wide range of temperature and frequency conditions by using only a small amount of material.

Many efforts have been made to unify test procedures in order to generate comparable and accurate test results. In 2000–2004, Rilem TC 182-PEB determined reproducibility of 10% for DSR measurements and provided relevant recommendations [2], which led to the development of the European Standard EN 14770 (2012). The latter is an overall guide for all DSR related issues, like rheometer specification and setup, sample preparation and test procedures. The recommended testing of the complex shear modulus ranges from 1 kPa to 10 MPa, while using a parallel plate with a diameter of 25 mm between 1 and 100 kPa and a diameter of 8 mm between 100 kPa and 10 MPa. According to EN 14770, appropriate temperatures should be chosen, while an equilibration time between 10 and 20 min should be applied to homogenise the temperature in the test specimen. For each temperature, oscillatory measurement with varying frequencies can be performed, considering the linear-viscoelastic (LVE) range of the material. This procedure is typically called frequency sweep or temperature sweep and still represents the standard test protocol.

However, the frequency sweep is time-consuming, as for each temperature an equilibrium time needs to be applied and latter requires shifting model to display master-curve. Other fields than paving, such as adhesive or tires, are using DSR in a different mode than frequency sweep. Thus, different approaches have been evolved so far for bituminous materials, based on non-stationary temperature conditions, where the temperature is continuously increased at a specific rate, designated as temperature ramp. During temperature ramping, continuous oscillatory measurements are performed at a fixed frequency in controlled-strain or controlled-stress modes. By running in different protocols, it can help evaluating the impact on DSR results, with different type of temperature conditioning. The applicability of different geometry setups are also compared together with the repeatability of the individual test protocols.

2 Experiment

Three bituminous binders were used, two non-modified binders, a soft, 70/100, and harder, 35/50, grades and a Polymer modified Bitumen (PmB) 25/55-55 as graded per EN 14023. Table 1 displays the basic properties of the three binders.

Table 1 Basic properties of the binders used in this study

Properties	Unit	35/50	70/100	PmB 25/55-55
Penetration value at 25 °C	0.1 mm	49	85	42
Softening point temperature	°C	54.8	45.8	58.4

Table 2 DSR test protocols and test parameters

Protocol	Lab	Type	Geometry diam/gap (mm)	Temperature	Frequency
Protocol 1	Lab1	F sweep	8/2	−10 to 40 °C, every 10 °C	0.1–10 Hz
			25/1	30 to 90 °C, every 10 °C	
Protocol 2	Lab1	T ramp 1.2 °C/min	25/1	20 to 90 °C	1.59 Hz (10 rad/s)
Protocol 3	Lab2	T ramp 3 °C/min	25/1		
Protocol 4	Lab3	T ramp 6 °C/min	10/2.5	−20 to 90 °C	

Four different test protocols, using DSR, were run by three different laboratories (not necessary using the same DSR machine): the conventional frequency sweep at different temperatures and three temperature ramping rates at fixed frequency, as summarised in Table 2. The design basis was to evaluate the impact of different protocols, which can optimise further test duration and data computation. It did not consider the variability induced by DSR machine, which will be evaluated and reported later.

Protocol 1 was a regular frequency sweep from 0.1 to 10 Hz at different temperatures according to EN 14470. The temperature step between each measurement was +10 °C with stationary temperature conditioning, for thermal and mechanical equilibrium. Two different diameters, 8 mm and 25 mm, together with gaps, 2 mm and 1 mm, respectively, were used, having an overlap at 30 and 40 °C. For each test temperature, suitable shear strains or stresses were chosen to ensure testing within the LVE-range. The full test setting for one replicate can last for a day and data needs further shifting model to build master curve.

For protocol 2, the temperature was continuously increased from 20 to 90 °C at a rate of +1.2 °C/min, according to German Standard DIN 52050 (2018) [3], which describes the test procedure for the Binder-Fast-Characterization-Test (In German: BTSV) [4]. During the temperature ramp, a continuous oscillatory measurement is performed at 1.59 Hz and 500 Pa shear stress. Values of the complex shear modulus and the phase angle are recorded once every second, demonstrating the temperature dependency. The test is aborted as soon as the shear strain reaches the threshold value of 100%, which corresponds to a complex shear modulus of 500 Pa. The test for one replicate last for about one hour.

For protocol 3, the temperature was continuously increased at a rate of 3 °C/min from +20 to 90 °C at a fixed frequency of 1.59 Hz (10 rad/s) in stress-controlled mode. Data were recorded with 10 points per °C. The geometry used was the one selected for high temperature with a 25 mm plate and a gap of 1 mm. The test run for about 30 min.

For protocol 4, the sample was fast cooled down to low temperature and then temperature was continuously increased by 6 °C/min from −20 to +90 °C at 1.59 Hz (10 rad/s). The geometry used was 10 mm plate and 2.5 mm gap. This enables to characterise various binders, including complex bitumen in a wide range of conditions [5]. The oscillatory measurement was performed first in stress-controlled mode with an applied torque of 10 μNm, until the strain achieved 10% value and then shifted automatically to strain-controlled mode. The test run for about 30 min for the whole temperature range.

3 Results

For each protocol, the measurements were performed on two replicates and results were analysed towards consistency. Later the mean values were used (geometric for shear modulus and arithmetic for phase angle). The analysis, presented here, was made by comparing the shear modulus and phase angle versus Temperature between −20 and +80 °C, at a frequency of 1.59 Hz (10 rad/s) for each binder. For the case of frequency sweep, only the data point at exact frequency was used without introducing any shifting factor. The four protocols are displayed in the same graph, with plain curves for Shear Modulus, and dotted lines for the phase angle.

For the 35/50, as shown in Fig. 1, the different protocols overlapped. For temperature above 20 °C, considering all four protocols, there is hardly any difference for the shear modulus and the phase angle. For temperature below +20 °C, only protocols 1 and 4 can be compared and a slight difference was recorded for the shear modulus.

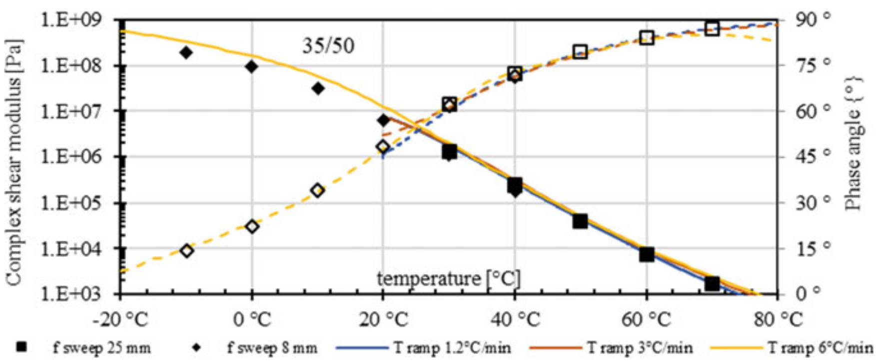

Fig. 1 Shear modulus and phase angle for 35/50 versus temperature, all protocols

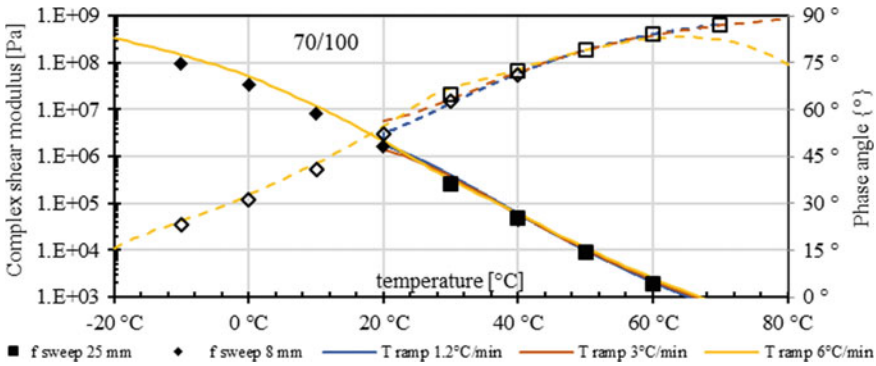

Fig. 2 Shear modulus and phase angle for 70/100 versus temperature, all protocols

As to the binder 70/100 in Fig. 2, a similar trend than for the binder 35/50 was observed, although for the phase angle at the highest temperature range a deviation for the protocol 4 was observed. However, it corresponded to a shear modulus below 10^3 Pa.

Lastly, for the PmB, in Fig. 3, the four protocols gave very similar results for both shear modulus and phase angle. For the latter, a characteristic plateau, from the polymer modification, was observed above +30 °C as compared to the standard paving grade.

Overall, between – 20 and +20 °C only two protocols can be compared; the shear modulus was slightly underestimated when protocol 1 was used. When compared to protocol 4, phase angles were comparable. For temperatures above +20 °C, the four protocols delivered comparable values in the same magnitude of range for shear modulus, although protocol 1 gave slightly lower values, and phase angle, regardless the binder.

Profound statistical analysis has been made for each binder and protocol in terms of repeatability and reproducibility. An example for the binder 35/50 is represented

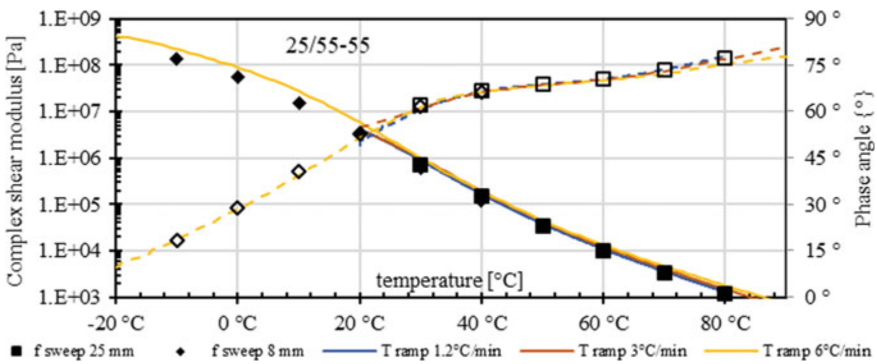

Fig. 3 Shear modulus and phase angle for 25/55-55 versus temperature, all protocols

Table 3 Comparative analysis of DSR measurements for 35/50 bitumen

Temperature	Protocol 1	Protocol 2	Protocol 3	Protocol 4	Average	StDev
Shear modulus (Pa)						
30 °C	1.3E+06	1.8E+06	1.9E+06	2.0E+06	1.7E+06	0.3E+6
40 °C	2.4E+05	2.7E+05	3.2E+05	3.0E+05	2.8E+05	0.3E+5
50 °C	4.0E+04	4.5E+04	5.1E+04	5.2E+04	4.7E+04	0.5E+4
60 °C	7.6E+03	8.2E+03	9.7E+03	1.0E+04	8.9E+03	0.4E+3
70 °C	1.7E+03	1.7E+03	2.2E+03	2.5E+03	2.0E+03	0.1E+3
Phase angle (°C)						
30	62.3°	60.4°	61.4°	62.0°	61.5°	0.8
40	72.6°	71.8°	71.3°	72.9°	72.1°	0.7
50	79.3°	79.0°	78.6°	78.9°	79.0°	0.3
60	84.1°	84.0°	83.6°	83.4°	83.8°	0.3
70	87.0°	87.0°	86.7°	85.1°	86.4°	0.9

below, for five temperatures between 30 and 70 °C. Table 3 displays the variation of the shear modulus and phase angle and average values obtained from the four protocols. For the shear modulus, with a standard deviation in range of 1/10 of the mean value, comparable to the value found in Rilem [2]. It did not show a systematic ranking between protocols, although the protocol 1 in frequency sweep had, for all temperatures, lower values by 20% as compared to the average values. For the phase angle, the reproducibility is rather good with a standard deviation below 1°.

4 Conclusion

The use of Dynamic Shear Rheometer for asphalt binder testing has increased in recent years. Different ways to run DSR have emerged based on different testing procedures. In this study, four different test protocols, one in frequency sweep at different temperatures and three at fixed frequency and different temperature ramping rates, were compared, considering three different bituminous binders.

The first analysis, comparing shear modulus and phase angle versus temperature, showed reasonable similar values between each protocol in a wide range of temperatures from − 20 to +80 °C. This is valid for plain bitumen and complex bituminous binder, such as Polymer modified Bitumen. An example of variability, for one bitumen and five temperatures, showed very good reproducibility for the phase angle measurement and a reasonable value for the shear modulus.

The computation of the data towards the most common DSR criteria, either PG critical temperatures, as per US standard, or temperature and phase angle at given shear modulus, as per European approach, will help determining the accuracy of running DSR in the most efficient way and will be reported in further publication.

However, this analysis does not count for variability coming from the machine itself or operating the test. One important question, that needs to be answered, is the effect of temperature equilibrium, as the main difference between frequency sweep and temperature ramping modes. This would be highly dependent, amongst other, on test device, on cooling/heating capacity and system. Further investigation are to be pursued, especially for complex bitumen such as highly modified ones.

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