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Measurement of Zero-Shear Viscosity in Asphalt

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Abstract: Zero shear viscosity (ZSV) is a widely accepted rheological parameter. Whereas previous work has mainly focused on unmodified bitumen, this paper discusses how to adopt ZSV for evaluating the rheological properties of polymer modified bitumen (PMB). We found that PMB was unable to reach the steady ZSV condition in a limited time even at a very low frequency level of 0.001rad/s, and the longer the recovery time, the more sufficiently the impact of viscosity could be eliminated. Our results also show that the multiple experimental recovery time test method is more reliable than the common creep test and recovery test for determining the ZSV of PMB. However, the impact of the delayed elastic strain and the delayed elastic recovery strain cannot be eliminated entirely. The ZSV obtained by the Cross model is between 10^2 and 10^3 times higher than those obtained by the other methods, the rest of which lead to similar results. Therefore, not only is accurate measurement critical when adopting ZSV to evaluate PMB, but a scientifically defined division between the viscous and elastic component is also important.

Key words: *Asphalt; Dynamic state tests; Measurement; Static state tests; ZSV.*

Introduction

Polymer modified bitumen (PMB) accounts for about 10% of the road bitumen market world-wide. In China, nearly 3 million tons are consumed annually, making it an important road construction material. A method for evaluating the high temperature properties of PMB is an important concern for successful application of PMB.

Zero shear viscosity (ZSV) is an alternative rheological parameter used to indicate high-temperature properties [1, 2]. Previous experiments have shown that the correlation between rutting and ZSV as measured by the Carreau model and by the Burgers model was 0.54 and 0.32, respectively [3]. Sybilski pointed out that the correlation coefficient between ZSV and rutting depth was the best indicator when compared with the correlation coefficient between other high-temperature parameters and rutting depth, especially for short time-aged binders. The correlation coefficient (r^2) between ZSV and rutting depth was more than 0.75, while the r^2 between other common parameters and rutting depth was only between 0.42 and 0.67 [4, 5]. Anderson found that the correlation coefficient between ZSV and road permanent deformation was very good, with an r^2 of 0.91 [6], by conducting 60°C hamburger rutting tests on stone matrix asphalt (SMA). Although rutting is affected by many other factors, the correlation coefficients between ZSV and the rutting depth are still meaningful.

In Europe, ZSV has been proposed as a high temperature specification parameter, while in the US, the National Cooperative Highway Research Program (NCHRP) 9-10 project has established a criterion based on parameters derived from Burger's viscoelastic model. Furthermore, the US regulatory agencies have concluded that mixing and compaction temperatures determined using ZSV at 3.0Pa.s and 6.0Pa.s meet the requirement of all asphalt mixtures [7].

Previous literature on ZSV has been mainly focused on unmodified bitumen rather than PMB. In this paper, we discuss how to obtain reliable ZSV for PMB, and we especially focus on methods used to separate the viscous component from elastic component.

Asphalt Rheological Characteristics and Zero-Shear Viscosity

From a rheology standpoint, properties like elasticity, viscosity, and plasticity are seen in every material, such as bitumen and bitumen mixtures, and all non-Newtonian fluid properties can be demonstrated by a comprehensive flow curve [8]. See Fig. 1.

As can be seen from this comprehensive flow curve, when the shear stress or shear strain rate is low, materials exhibit Newtonian fluid characteristics. With increasing shear strain rate, materials show increasing false plastic flow and finally present Newtonian flow properties once again. In order to show a material's false plastic properties at high shear rate and Newtonian properties at low shear rate, Carreau put forward an equation to describe the rule of material viscosity behavior in 1979. Fig. 2 shows the physical meaning of Carreau's equation parameters.

$$\eta_{\mathrm{a}} = \eta_{\infty} + (\eta_0 - \eta_{\infty})\left[1 + (\lambda D)^2\right]^{\frac{c-1}{2}} \qquad (1)$$

where, η_a = Viscosity, η_0 = Zero-Shear Viscosity, η_∞= infinitude shear viscosity, λ = constant, D = shear rate, and c = constant.

Fig. 2 shows the curve of viscosity versus shear rate plotted on a logarithmic scale for bitumen and other non-Newtonian materials. With a decrease in shear rate, the bitumen viscosity will finally reach a maximized constant value called ZSV, an asymptotic viscosity value when the shear rate approaches zero. When the shear rate increases to a critical point, $\lambda D > 1$, the viscosity will start to decrease, which is called "shear thinning". If an extremely high shear rate is reached, $\lambda D >> 1$, viscosity will reach the second Newtonian

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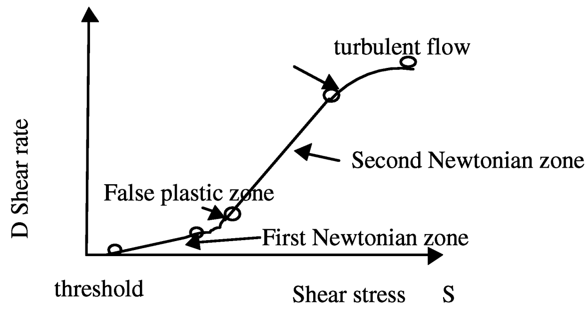

Fig. 1. Sketch Map of Comprehensive Flow Curve.

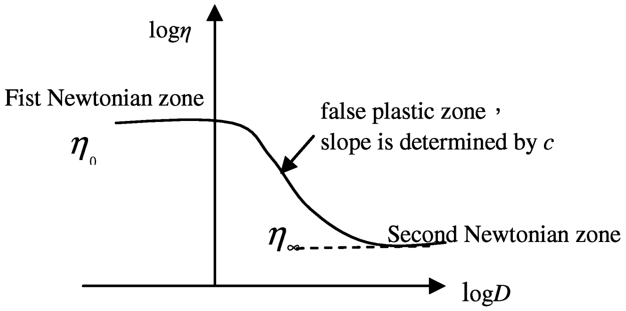

Fig. 2. Curve of Viscosity versus Shear Rate.

zone, where η_∞ is smaller than η_0 and $\eta_a=\eta_\infty$.

Measurement Methods for Zero-Shave Viscosity

ZSV Measurement Methods via Static State Tests

ZSV can be measured by two kinds of approaches, namely static and dynamic state testing. Because of the viscoelastic behavior of bitumen, the real ZSV can only be obtained after separating the viscous flow component from the delayed-elastic strain component. Commonly used static approaches are described as follows:

1) ZSV can be calculated from the slope of a creep curve obtained from a sufficiently long duration creep test.
2) ZSV can be estimated from remaining strain and recovery time from a sufficiently long duration creep recovery test.
3) ZSV can be fitted by a disperse delayed spectrum.

ZSV Measurement Methods via Dynamic State Tests

ZSV can be obtained via the slope of a creep curve from a single cycle creep test, which is always time-consuming if allowed to reach a steady flow state and complete recovery (a few hours or a few days). By using frequency sweeps with periodic sine changes and low frequency loading, when the angular frequency approaches zero, the viscosity can be approximately taken as the ZSV. ZSV can also be calculated by the special software Rhea [9]. Additionally, a dynamic shear rheometer, which is becoming increasingly popular in China, makes it possible to determine ZSV by the dynamic method. Commonly used dynamic state test methods include:

1) ZSV converged by either low frequency dynamic test or frequency sweep test.
2) ZSV fitted with correlative models, such as the Cross model or the Carreau model.

Measurement of Zero Shave Viscosity of Modified Asphalt Binders

ZSV Measurement via Creep Experiment

ZSV can be measured using a creep experiment with the creep recovery coming mainly from delayed elastic strain. The delayed elastic strain will recover completely after a long enough recovery time, and the remaining deformation is only contributed to by viscous flow. This paper focuses on measuring the ZSV of PMB, which is much more complicated than measuring the ZSV of unmodified bitumen.

The ZSV of two kinds of Styrene-Butadiene-Styrene (SBS) modified bitumen binders were measured by an AR2000 dynamic shear rheometer produced by the AT Company in the U.S.A. The high temperature performance grade for one kind of modified bitumen is 76, and for the other it is 82; as a result, the asphalts were named PG76 and PG82, respectively. The nonlinear properties were taken into consideration, and the experiment time was extended to 3hrs loading/3hrs unloading and 5hrs loading/5hrs unloading at 64°C, respectively. The creep stress was set at 30Pa. The ZSV obtained by three methods is shown in Table1.

Table 1 indicates that the ZSV obtained from recovery behavior is lower than that from creep behavior. The results obtained from a delayed disperse spectrum model fitting are close to those obtained from creep. The ZSV obtained from a 5hrs creep is greater than that from a 3hrs creep. This indicates that a steady viscous flow state is very difficult to achieve in a limited time. The creep strain measured includes both the viscous flow response and the delayed elastic response; due to the delayed elasticity, it could not entirely recover. Therefore, these approaches are not useful for measuring the ZSV of PMB.

In order to allow the delayed elasticity strain to recover sufficiently, multiple recovery time experiments with one second creep and nine seconds recovery were adopted to separate viscosity from elasticity. The strain after one second creep is noted as ε_L, while the remaining strain after a nine second recovery is noted as ε_P. Table 2 shows the test results of two modified asphalt binders of PG76 and PG82. As shown in the table, the stress magnitude influences the results, and the data of $\varepsilon_L/\varepsilon_P$ indicated that the ε_P obtained from the test method of nine seconds multiple recovery

Table 1. Zero Shear Viscosity Obtained from Curve Fitting.

Modified Asphalt		Zero Shear Viscosity η_0, Pa · s			
		PG76		PG82	
Load-unload Time		3hrs-3hrs	5hrs-5hrs	3hrs-3hrs	5hrs-5hrs
Test Methods	Creep	1.00×10^5	1.72×10^5	3.93×10^5	4.57×10^5
	Recovery	8.00×10^4	9.31×10^4	3.07×10^5	3.53×10^5
	Disperse Delay Spectrum Model Fitting	1.33×10^5	1.69×10^5	4.09×10^5	4.47×10^5
	Standard Deviation of Model Fitting	1.13	0.4654	1.04	0.81

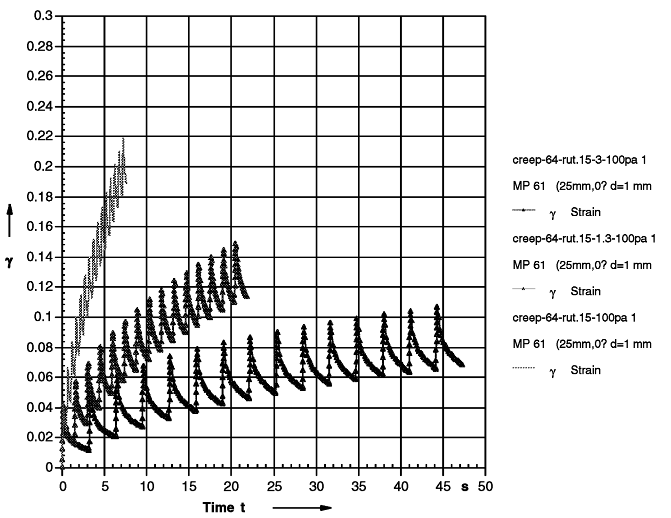

Fig. 3. Creep Recovery Response Comparison of PG76 at 0.15s Loading Time Followed by 0.35, 1.3, and 3s Unloading Time.

Table 2. Results of Multiple Recovery Time Test.

Asphalt	Stress, Pa	ε_L	ε_P	$\varepsilon_L/\varepsilon_P$	η_0, Pa · s
PG76	30	0.03960	0.02084	1.90	12956
	100	0.13408	0.06773	1.98	13366
	300	0.39736	0.19245	2.06	14029
PG82	30	0.02425	0.00449	5.40	60133
	100	0.09890	0.01836	3.10	49010
	300	0.25077	0.06402	3.92	42174

time is less than the ε_L obtained from the test method of one second creep loading. This indicates that the PMB exhibits non-Newtonian proper- ties under the above mentioned test conditions. Even when the mul- tiples of recovery time were increased to 0.15s loading time follow- ed by 0.35, 1.3, and 3s unloading, the delayed-elastic strain and de- layed-elastic strain recovery of PMB could not be eliminated, as shown in Fig. 3. With the increase in time multiples, the viscous component was separated further. Although it cannot result in a reliable ZSV, the multiple recovery time tests is more practical than the common creep test and recovery test.

Measurement of ZSV by Frequency Sweeps

To avoid a time-consuming creep recovery test, ZSV can also be determined by dynamic frequency sweeps. ZSV were measured by frequency sweeps using an AR2000 dynamic shear rheometer with a parallel plane 25mm in diameter, frequency levels ranging from 0.1 to 100rad/s, and strain levels of 10% and 1%. In order to explain the effect of additives on mechanical behavior of asphalt binders, parallel tests on both unmodified bitumen and PMB were done. The viscous response of unmodified bitumen can reach steady state quickly, indicating steady Newtonian flow properties. The model-fitting procedure was used to fit the data, yielding ZSV values of 246.9Pa · s at 64°C and 109.1Pa · s at 70°C.

The test results of the PG82 PMB presented in Fig. 4 show that the steady ZSV state is difficult to obtain, and the phase angle δ decreases with decreasing frequency, indicating that the elastic component becomes greater and the storage modulus becomes lower with decreasing frequency. The storage modulus is larger than the loss modulus at the low frequency level, and the elastic response exceeds the viscous response. From these experiments, one can conclude that, although different PMB have different rheological properties, none can obtain a steady ZSV, even if the frequency is as low as 0.001rad/s.

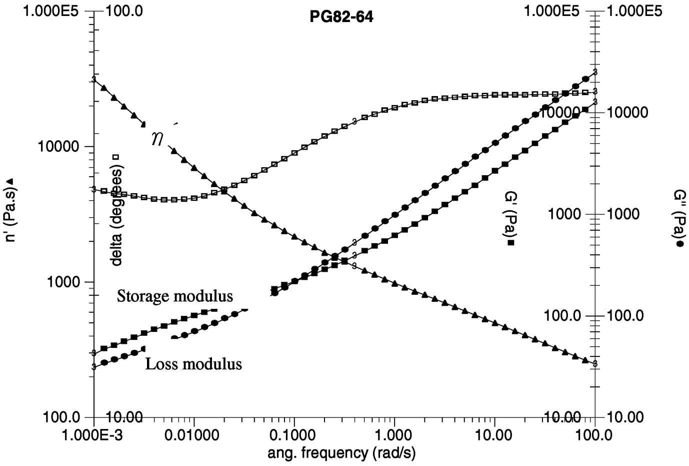

Fig. 4. Frequency Sweep of Modified Asphalt PG82.

Even at low frequency levels or at low shear rates, the viscosity of PMB does not tend towards a constant value. The correlation between the data obtained from long term creep testing and that obtained from frequency sweep testing was a concern. As a kind of viscoelastic material, the mechanical behavior of bitumen is determined by the ratio of recovery time and test time. In order to expeditiously compare the results obtained from different test methods, frequency sweep time should be the same as shear time in the creep test. The relation of the two parameters is $\omega = 1/t$, where the unit of ω is radians per second, and ω is angular frequency in the dynamic state. Shearing in a 3hrs creep test is equal to a 9.3E-5rad/s frequency level, and shearing at 5hrs is equal to a 5.5E-5rad/s frequency level. The results of the two test methods are shown in Figs. 5 and 6. The fitting curves indicate that the correlation of the results obtained from the two methods is very good. Viscosity does not tend towards a steady state at the range of the test frequency. For these asphalt binders, results obtained from

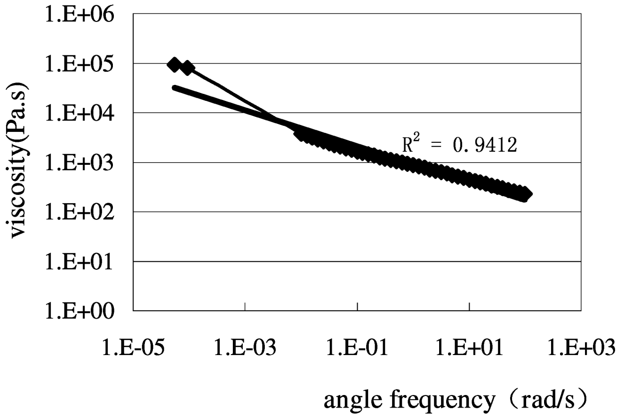

Fig. 5. Viscosity of PG76 from Creep Test and from Frequency Sweep.

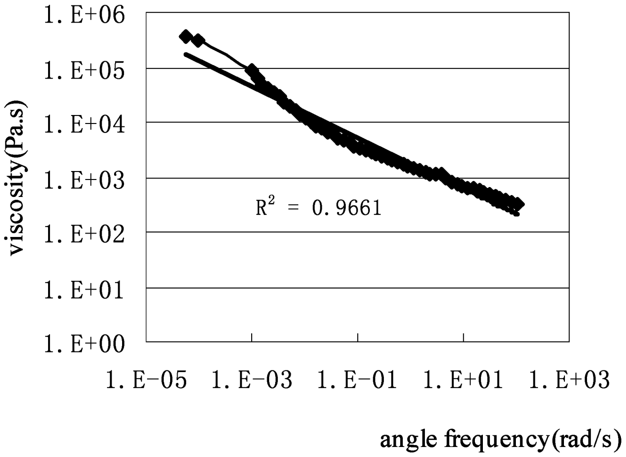

Fig. 6. Viscosity of PG82 from Creep Test and from Frequency Sweep.

Table 3. ZSV Comparison from Various Fitting Model.

Fitting Model	PG76	PG82
ZSV by Cross Model (Pa·s)	2.33×10^7	2.21×10^8
ZSV by Carreau Model (Pa·s)	5.72×10^4	5.50×10^5

creep are above the trend lines, which indicate that the results obtained from creep are higher than those from frequency sweeps.

From the test data, one can conclude that the viscosity of SBS-modified bitumen is 10^3 to 10^4 times higher than the viscosity of unmodified asphalt, and the ZSV state is difficult to obtain within the range of the test frequency. Therefore, the ZSV can be determined by rheological theory and empirical fitting expressions, such as the Cross model and the Carreau model. The fitting results of PG-76 and PG82 are listed in Table 3. ZSV, as determined using the Cross model, is much higher than the ZSV resulting from the Carreau model [10].

The data in Tables 1 and 3 indicate that ZSV determined by the Cross model is the highest, between 10^2 to 10^3 times higher than the others. The second highest is the ZSV determined by creep and the delayed disperse spectrum, and the lowest estimate is given by recovery and the Carreau model, which are similar. The Carreau model was recommended for calculation of ZSV by [11] after they compared 15 rheological models.

Conclusion

The following conclusions were drawn from this study:

1. PMB cannot reach the steady viscous flow state in a limited time. The creep strain measured includes not only the viscous flow response, but also the delayed elasticity response. Furthermore, the remaining recovery strain consists of not only viscous flow strain, but also the delayed elasticity recovery strain.
2. The multiple recovery time experiment is more reliable than either the common creep test or the recovery test for determining the ZSV of PMB, although the impact of the delayed elastic strain and the delayed elastic recovery strain cannot be eliminated entirely.
3. The results of the frequency sweep indicate that PMB cannot reach a steady ZSV condition in a limited time, even at the very low frequency level of 0.001rad/s. The longer the recovery time is, the more sufficiently the impact of viscosity could be eliminated. This is quite different from the behavior of unmodified bitumen, which can achieve steady state very quickly even at a relatively high frequency level of 0.1 to 1rad/s.
4. Different modes lead to different ZSV. The ZSV obtained by the Cross model is the highest, 10^2 to 10^3 times higher than those obtained by other models. The second highest is ZSV measured by creep and disperse delayed spectrum, and the lowest ones are ZSV as measured by recovery and the Carreau model, which are similar.
5. Accurate measurement is not the only critical parameter when adopting ZSV to evaluate PMB, and a scientific division between the viscous component and elastic component is also important.

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