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High Temperature Rheological Performance of Graphene Modified Rubber Asphalt

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Abstract

To elucidate the high temperature rheological capability of graphene modified rubber asphalt， three contents of graphene and crumb rubber were prepared by a combination of mechanical agitation and high speed shearing machine ,then used dynamic shear rheological test (DSR) and multiple stress creep recovery (MSCR) tests to evaluate. The hardness and softening point with rotational viscosity of samples raised with the addition of graphene, especially the addition of 0.04%. Dynamic shear rheological test revealed that the dynamic shear modulus G*, rutting factor G*/Sin δ, and zero shear viscosity (ZSV) of graphene-modified rubber asphalt were greatly influenced along with graphene-increased, on the contrary, phase angle δ which characterize the viscoelastic ratio of asphalt decreased. Multiple stress creep recovery (MSCR) tests showed that the graphene-enhanced rubber asphalt had high-temperature stability through non-recoverable creep compliance (Jnr). Based on these findings, graphene-modified rubber asphalt binders with the addition of 0.04% graphene had good viscoelastic properties as well as high temperature rutting resistance performance. In the meantime, G*/Sin δ, ZSV, and Jnr100, Jnr3200 have good correlation, which can reveal the excellent high-temperature stability performance of asphalt.

Keywords: *Graphene; High Temperature rheological properties; MSCR; Zero shear viscosity; Rutting*

1 Introduction

Rutting is caused by the accumulation of irreversible and permanent deformation for the repeated heavy wheel loadings on the various pavement layers under High-temperature especially, it is one kind of the main damaged of pavement at present. The deformation of rutting is a serious safety hazard to vehicles because hydroplaning can occur in the presence of rutting in rainy weather, setting off serious traffic accidents, and it has been taken into independent detection index of highway technical conditions account. Therefore, asphalt pavement designs and constructions are the most important to prevent the occurrence of rutting deformation problems [1-2]. The structure stability of asphalt pavement is correlate to the normal use of transportation, and the use of high-quality modified asphalt is one of the key measures to solve the problem of pavement stability, so the development of modified asphalt with excellent performance has important practical significance for the construction of asphalt pavement [3-5].

Multiple stress creep recovery (MSCR) tests provide a reasonable basis for construction of high temperature [6-7] ,which in a tropical climates and repeated heavy wheel loadings on asphalt pavement, therefore, MSCR is used to evaluate the rutting resistance of asphalt under different stress levels, which characterized by non-recoverable creep compliance Jnr[8]. Dynamic shear rheology test (DSR) can overcome limitations when measures asphalt binders, rheological properties in a wide scale of temperatures and frequency, based on rheological in many previous studies, the rutting resistance can be used to show the high temperature performance of asphalt pavement [9-10].

Graphene is a kind of nanomaterial with high-tech and wide application potential, it has been used as an asphalt modifier in practical engineering for its excellent properties [11]. Many studies [12-14] show that the additive of graphene enhances the miscibility of modifiers in asphalt binders, increases its high temperature performance of modified asphalt with elasticity and rutting resistance properties. However, there are few studies on the dynamic rheological properties of graphene-modified asphalt, so this paper conducts research on it

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In this paper, the high temperature rheological performance of graphene modified rubber asphalt was valuated using dynamic temperature, frequency scanning and MSCR tests by dynamic shear rheometer, encouraging to the practical engineering application of graphene-modified rubber asphalt.

2 Materials and Methods

2.1 Materials

The base asphalt used in this study is Maoming AH-70. The crumb rubber powders with 40 mesh with a measured density is 1.12g/cm^3 and there is no apparent impurity and it is provided by Changzhou Jingcheng Chemical Co Ltd, China. Graphene is a single-layer graphene (SSA: 50-200 m^2/g, carbon content is 98%) which is purchased from Shenzhen Turing Evolution Technology Co., Ltd， and the relevant technical indicators of graphene are shown in Table 1:

Table 1 Graphene-related parameters

Test metrics	Unit	Technical requirements	Result
Bulk density	g/ml	0.01-0.02	0.013
Carbon content	%	—	98
Specific surface area	m^2/g	50-200	185
water content	%	＜2	1.3
Monolayer rate	%	＞80	95

2.2 Preparation of graphene modified rubber asphalt binders

The contents of graphene (0%,0.02%,0.04% of original asphalt) modified rubber asphalt binders were prepared by glass rod and high shear mixer to achieve homogenous dispersion of the graphene and rubber in asphalt binders, and the preparation method followed: The base asphalt was heated to the melting flow state in an oven under 135 ℃ and the weight of 300 g was melted into a container, a layer of the asbestos net is laid on the heating furnace. The melting asphalt container was heated to keep the glass bar stirred continuously and the temperature of asphalt was recorded with a thermometer at any time. Keep the asphalt temperature 175 ℃ and stir for 10 minutes. The crumb rubber powder and graphene were prepared in advance then mixed into the asphalt and stirred successively until all the modifiers were immersed in the asphalt. Then a high-speed shearing machine was used to rotate for 1 hour at a speed of 5000r/min, then the mixture was kept in static condition for 1.5h below 175℃. The preparation process of graphene modified rubber asphalt binders is illustrated by a flowchart as shown in Figure 1.

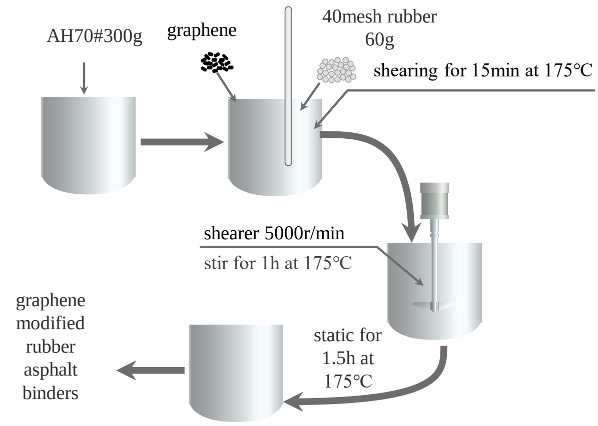


Figure 1 The preparation process of graphene-modified rubber asphalt binders

2.3 Experimental Methods

2.3.1 Physical properties tests

Penetration, softening point, and rotational viscosity are used to be the three main index to value high temperature performance of asphalt binders, which followed the standards of JTG E20-2011 T0604-2011, T0604-2011, and T0625-2011method.

2.3.2 Dynamic temperature sweep test

DSR test of graphene-modified rubber asphalt was carried out with Bohlin CVOR-ADS dynamic shear rheometer which produced by Malvern Instrument Company. The strain is controlled by 2% with a fixed frequency at 10rad/s. The dynamic temperature of graphene-modified rubber asphalt was obtained by temperature sweep at 46-82℃ (with an interval of 6℃). Rutting factor (G*/Sin δ), Phase angle, and Dynamic complex modulus of samples were measured under the linear viscoelastic region of asphalt binders.

2.3.3 Dynamic frequency sweep test

To analyze the performance of high temperature viscoelasticity parameters of graphene-modified rubber asphalt, the Dynamic frequency sweep test selected the temperature at 64 ℃ as data analysis basis, the frequency was set to 0.1Hz～10Hz, and the strain was controlled to 2%.

2.3.4 Multiple stress creep restoration test (MSCR)

THE MSCR test is carried out through the dynamic shear rheometer, and stress levels are fixed in 100Pa ，3200Pa at 82℃. One second shear creep load is applied on the sample, following by nine-second recovery, and then ten cycles of creep and recovery were conducted under the shear load.

3 Results and Discussions

3.1 Physical properties tests

Physical property tests which consisted of penetration, softening point, and viscosity at 135℃ [15-16]

were conducted on three contents of graphene-modified rubber asphalt binders according to JTG E20-2011. The test results are shown in Table 2:

Table 2 Physical properties of three contents of graphene-modified rubber asphalt binders

Different dosages of graphene	Penetration 25℃ (0.1mm)	Softening point (℃)	135℃ Rotational viscosity (Pa• S)
0%	43.1	69.3	7.39
0.02%	44.5	79.4	7.46
0.04%	41.2	82.4	9.89

The change of viscosity owning to graphene addition in the rubber asphalt (135℃) is shown in Table 1. It can be found that the viscosity of rubber asphalt with graphene is significantly higher than that of rubber asphalt. It can be concluded that the addition of graphene increases the viscosity of rubber asphalt and enhance the resistance of rubber asphalt to external forces to a certain extent. Obviously the viscosity test showed that graphene modified rubber asphalt has better rutting resistance, excessive viscosity will make asphalt consume more energy during the pumping and mixing, and emitting more greenhouse gases, which is not conducive to environmental protection. In the softening point test, the joining of graphene can improve the softening point and the high temperature stability, comparing to the rubber modified asphalt in the certain extent [17]. Penetration is an important index to evaluate the physical properties of asphalt. In this test, when graphene content is 0.04%, the hardness of modified asphalt was higher, which indicated that it was stable at high temperatures.

3.2 Dynamic temperature sweep test

Dynamic shear rheometer is a basic test instrument of viscoelastic materials. In the pavement performance specification of asphalt, SHRP pointed out that in the process of strain control, the swing plate rotates at specified frequency to complete a specified period, and the same time ,required torque, shear stress, shear strain, and phase angle are measured [18]. The shear stress S, shear strain D, dynamic complex modulus G*, and phase angle of asphalt specimens are calculated as follows:

$$S = \frac{2\mathrm{T}}{\pi r^3} \tag{1}$$

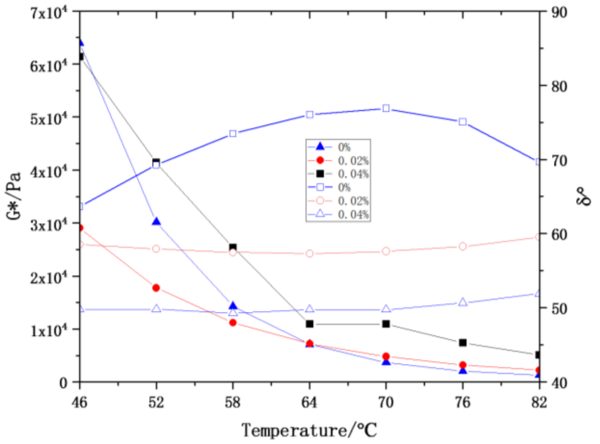
$$D = \frac{\theta \cdot \mathrm{r}}{\mathrm{h}} \tag{2}$$

$$G *= \frac{S_{max} - S_{min}}{D_{max} - D_{min}} \tag{3}$$

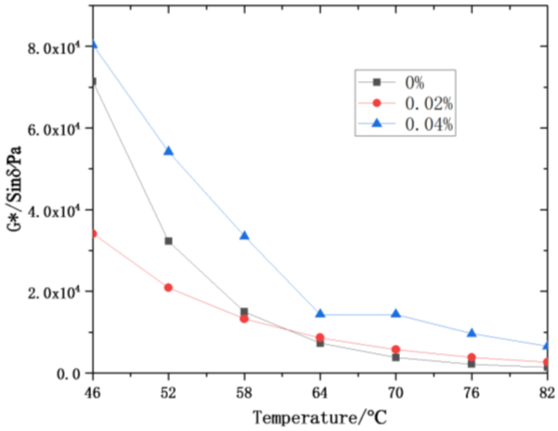
$$\delta = 2\pi \mathrm{f} \cdot \Delta \mathrm{t} \tag{4}$$

In the formula, T is the maximum torque; R is the radius of the swing plate (12.5mm), h is the height of the specimen (1mm); theta is the rotation angle of the swing plate; S_{max}, S_{min}, D_{max}, D_{min} stands in for the maximum or minimum shear stress and strain that the specimen bears; and t is the lag time.

There is little doubt about that performances of pavement mainly depending on the viscoelastic and mechanical behavior of asphalt. Until now, the complex modulus G*, phase angle δ, and rutting factor G*/Sin δ of three modified asphalt binders at different temperatures were measured by dynamic shear rheological test to characterrize the high temperature performance [19-20]. The results of G*, δ and G*/Sin δ of modified asphalts with temperature is shown in Figure 2:



(a) Complex modulus -temperature- phase angle relationship of different modified asphalt



(b)Rutting factor -temperature relationship of different modified asphalt

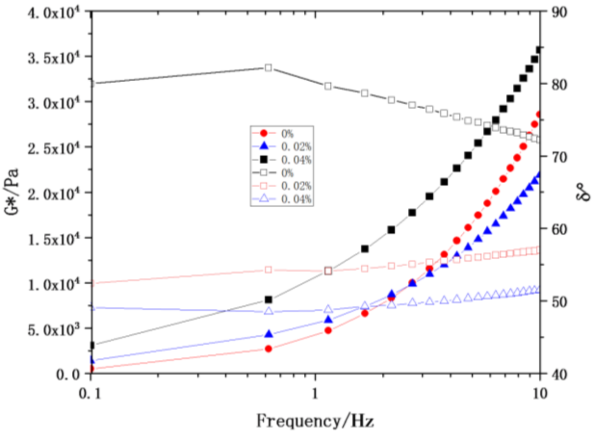
Figure 2 The result of dynamic temperature sweep test.

In terms of the research of SHRP, the higher temperature, the anti-rutting performance of asphalt worser, it is well accepted that improving the anti-rutting performance of asphalt is very important [22]. Figure 2(a showed that the complex modulus G*, rutting factor G*/Sin δ are obviously related to temperature. With the increasing of temperature, the rutting factor G*/Sin δ decreases gradually. At relatively low temperature, rubber asphalt with 0.04% graphene content is larger than rubber asphalt with 0.02%, 0%graphene addition. After the temperature is higher than 64℃, the addition of graphene enhance Dynamic complex modulus G*, Rutting factor G*/Sin δ of rubber asphalt. When the G*/Sin δ bigger, the permanent deformation of asphalt binder decrease due to energy dissipation. It showed that graphene-modified rubber asphalt binders have better rutting resistance and stability at high temperatures [23].

Phase angle δ can be used to characterize the viscoelasticity of asphalt [24]. In Fig 2, it is obvious that the fluctuation of phase angle of graphene modified rubber asphalt is gentle with the increase of temperature, and the addition of graphene reduces the phase angle with rubber modified asphalt, which indicates that the modified asphalt binders have good resilience.

3.3 Dynamic frequency sweep test

The loading mode of dynamic frequency sweep following the time-temperature equivalence principle is used to ensure the test conditions and samples meet the linear viscoelastic theory at the same temperature [25]. The changes in dynamic complex modulus and phase angle of modified asphalt were observed at different frequencies. The experimental results are shown in Figure 3:



(c)Complex modulus -frequency- phase angle relationship of different modified asphalt

Figure 3 The results of the dynamic frequency sweep test

The results of dynamic frequency sweep test showed that the complex modulus of modified asphalt increases gradually with the frequency raising, and the dynamic complex modulus of graphene content of 0.04% showed obvious advantages. From the changed of phase angle, the phase angle of graphene modified asphalt binders increase to a certain extent with the frequency raising, which shows that the viscoelastic component of graphene modified asphalt increased and the elastic component decreased gradually. However, compared with rubber modified asphalt, the elastic property of graphene modified asphalt binders is larger, which indicated that graphene modified asphalt resisted external force deformation at high temperatures.

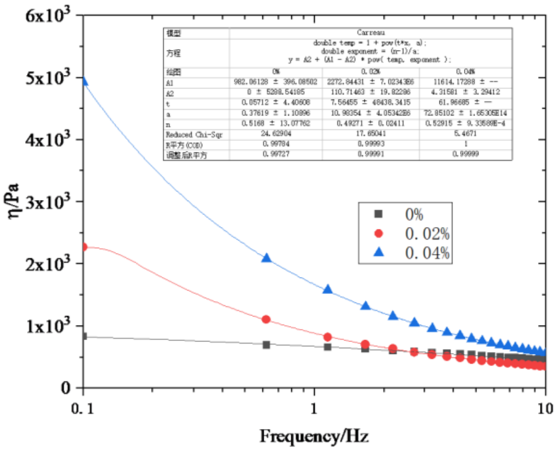
Correlation studies have proclaimed that ZSV can be used as an key index of high temperature performance of asphalt [26-27]. ZSV refers to the viscosities directly measured or calculated at a certain temperature when the shear rate reach or approaches zero, so it can be used to characterize the viscous performance of materials, it is an inherent property of materials, also known as absolute viscosity [28]. Rutting deformation at high temperatures is a slow process, so it is reasonable to use zero shear viscosity be a high temperature performance evaluation of graphene-modified rubber asphalt binders with different content. ZSV can be evaluated by dynamic frequency loading test. In this paper, the Zero shear viscosity of modified asphalt at 64℃ was predicted by using Carreau, Cross, and other related models. The Carreau and Cross equations were fitted as follows:

$$\eta_a=\eta_\infty+（\eta_0-\eta_\infty）[1+(\lambda D)^2]^{\frac{C-1}{2}} \tag{5}$$

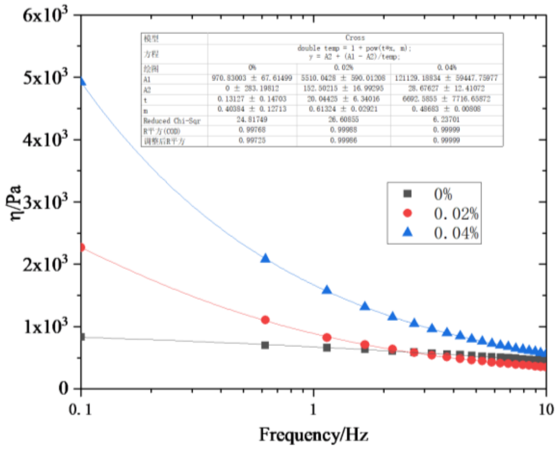
$$\eta_a=\eta_\infty+\frac{\eta_0-\eta_\infty}{1+（k^*D）^P} \tag{6}$$

In the above formulae, D is the shear rate，when D →0，there is η a → η0，which is zero shear viscosity，when D→∞，η a → η∞，shear viscosity, that is, infinite shear viscosity.

According to the fitting results of the experiment, as shown in Figure 4:



(d) Fitting curves based on the carreau model



(e) Fitting curves based on Cross model

Figure 4 Fitting results of zero shear viscosity

Figure 4 shows that with the frequency raising, the complex viscosity of the three different modified asphalts decreases gradually, and there are obvious differences, it can be concluded that this is because of the addition of graphene enhanced viscosity with modified asphalt. Among them, when the content of graphene reach 0.04%, the complex viscosity is increasing markedly, it can be seen from this graphene improved the viscosity with the modified asphalt to a certain extent. ZSV of modified asphalt was obtained by two fitting methods. The calculation table is as follows: Table 3and Table 4.

Table 3 Carreau fitting parameter results

Different dosages of graphene	η_0	Correlation coefficient
0%	982.1	0.99
0.02%	2272.8	0.99
0.04%	11531.3	1

Table 4 Cross fitting parameter results

Different dosages of graphene	η_0	Correlation coefficient
0%	970.8	0.99
0.02%	5519.6	0.99
0.04%	121878.8	1

Table 3 and 4 show that with the addition of graphene , the ZSV of rubber modified asphalt increase gradually. Moreover, the Zero shear viscosity of graphene modified rubber asphalt with the dosage of 0.04% graphene content was obviously higher than that of the other two additions. The result shows that the rutting resistance of asphalt is obviously enhanced with the addition of graphene, which was beneficial to the high-temperature performance remarkably which had a good correlation with the rutting factor.

3.4 Multiple stress creep recovery (MSCR) tests

MSCR uses the delayed elastic recovery performance of asphalt under applied stress levels to evaluate the high temperature performance of asphalt, and the cumulative strain has a good correlation with the high temperature performance of the mixture [29-30]. Compared to the G*/Sin δ of sinusoidal loading pattern, asphalt will produce creep compliance under stress. After stress is removed, some of creep compliance will be recovered while non-recoverable creep compliance will accumulate to the next load-deformation accumulation, which is the special feature of viscoelastic materials [31]. The pavement under repeated loading and unloading of vehicle load is also a process of cumulative deformation, so the MSCR can simulate the asphalt pavement strain accumulation process truly and accurately. Different from the traditional PG grading requirements, MSCR reflects the cumulative deformation of asphalt under repeat loading and unloading conditions, and the loading pattern is not affected by asphalt deformation [32].

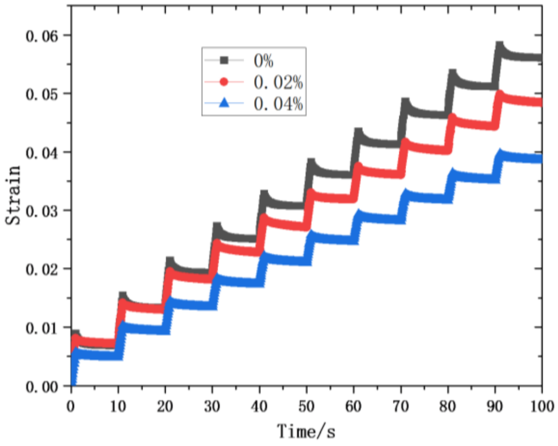
Jnr and Jnr-diff under different levels of stress are calculated according to the strain collected. The calculation methods are as follows:

$$Jnr=\frac{\varepsilon_{r-\varepsilon_0}}{\delta}\times 100\% \tag{7}$$

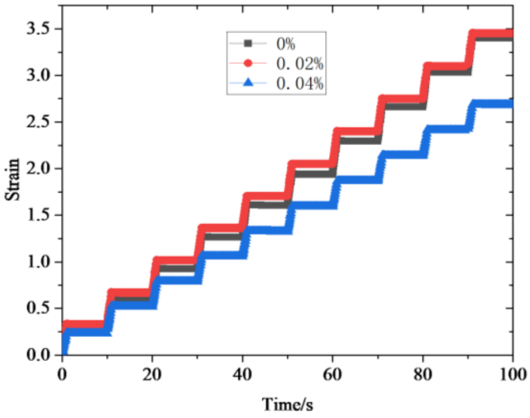
$$Jnr\text{-diff}=\frac{J_{nr(3200Pa)}-J_{nr(100Pa)}}{J_{nr(3200Pa)}}\times 100\% \tag{8}$$

Where the ε_0 and ε_r represent the initial deformation and residual deformation after 9s Restoration, the “δ” represent shear stress, J_{nr} represents non-recoverable creep compliance, while the $J_{nr\text{-diff}}$ represents the rate of increase in J_{nr} value when the stress changes from 100Pa to 3200Pa.

Figure 5 show the MSCR test results of three different kinds of graphene modified rubber asphalt under two stress levels. The horizontal ordinate is the whole time of the test, and the longitudinal coordinate is the strain change of the modified asphalt.



(f) Time-strain relationship of different modified asphalt (100Pa)



(g) Time-strain relationship of different modified asphalt (3200Pa)

Figure 5 The results of multiple stress creep recovery (MSCR) tests

According to Figure 5, non-recoverable compliance values at 100Pa and 3200Pa stress levels can be obtained as shown in Table 5.

Table 5 100 Pa and 3200 Pa stress levels for non-recoverable compliance values

Different dosages of graphene	J_{nr100}(KPa)	J_{nr3200}(KPa)	$J_{nr\text{-}diff}$ (%)
0%	0.56	1.06	47.2
0.02%	0.48	1.08	55.1
0.04%	0.38	0.84	54

The results of the MSCR test show that Jnr at 3200Pa is larger than that at 100Pa, which indicated that with the creep load level increasing , the asphalt maximum strain level also increase. This is mainly due to the higher permanent deformation and lower percent recovery during the creep loading level increase Under two stress levels, asphalt with a graphene content of 0.04% has a smaller Jnr. It could be seen that the content of graphene decrease the viscous deformation of asphalt and had better high temperature deformation resistance, which had a good correlation with the rutting factor and zero shear viscosity mentioned above.

Compared with ordinary rubber asphalt, the rate of change of non-recoverable creep stress with the time of graphene-modified rubber asphalt $J_{nr\text{-}diff}$ improved with the increase of graphene addition, shows that although the higher content of graphene, it had better high-temperature deformation resistance, but it's viscous deformation was more obvious since the shear stress.

4 Conclusion

40 mesh crumb rubber powder and single-layer graphene were used as asphalt modifiers in this study, the high temperature performances of different addition of graphene modified rubber asphalt were characterized by using DSR and MSCR, and variance analysis was verified feasibility through J_{nr} and $J_{nr\ \text{-}diff}$ to evaluate high-temperature performance of graphene modified rubber asphalt. Based on this limited laboratory survey, the following conclusions are shown:

(1) According to the results of the dynamic temperature sweep test, adding graphene to modified rubber asphalt leads to the increase of rutting factor, which improve the high temperature performance of asphalt lead to a higher working temperature range.

(2) In the DSR test, it was obvious that the addition of graphene made the modified rubber asphalt have better deformation resistance and elastic recovery ability at high temperatures. With the temperature raising, the rutting resistance of rubber asphalt with 0.04% addition of graphene was the best. Zero shear viscosity (ZSV) of modified asphalt is predicted by fitting Carreau and Cross models. It was found that the addition of graphene significantly improved the rutting resistance of asphalt and the high temperature performance of asphalt.

(3) The strain response and delayed elasticity of rubber asphalt and graphene modified rubber asphalt were observed by the MSCR test. The results show that the high temperature performance of graphene modified rubber asphalt is greatly affected by the stress level. According to the non-recoverable creep compliance (Jnr) obtained from the MSCR test, it could be found that the rutting resistance of graphene modified rubber asphalt is significantly improved compared with rubber asphalt. Generally speaking, it shows that the positive effect of graphene content on permanent deformation is 0.04%.

(4) The process of residual deformation is caused by repeating loading and unloading in multiple stress creep recovery test (MSCR) had a great extent correlation with the process of rutting was caused by vehicle loading at a certain temperature.

For the application of graphene to modified asphalt pavement, relevant demonstration engineering applications have been carried out, but further exploration and research are still needed in the macro and micro properties of graphene-modified asphalt, so as to improve the development requirements of road durability.

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