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Investigation on high-temperature performance of waste-based high-viscosity asphalt binders (WHABs) by Repeated Creep Recovery (RCR) test

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2 **high-viscosity asphalt binders (WHABs) by Repeated Creep**
3 **Recovery (RCR) test**
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Abstract

This study reviewed and compared the test methods for the high-temperature performance of asphalt binder, and selected Repeated Creep Recovery (RCR) test to evaluate the high-temperature performance of three waste-based high-viscosity asphalt binders (WHABs, including D-I, D-II and F) and a control sample (TAFAK-SUPER modified asphalt, TMA). Meanwhile, shear stress, test temperature, and aging degree of binders, which could influence high-temperature behavior of binders, were also investigated, and related evaluation indexes including recovery compliance (R), non-recoverable creep compliance (J_{nr}), and viscous stiffness modulus (G_v) were compared. Results show that WHABs has much better high-temperature performance than TMA; shear stress has the most prominent effect on binders' high-temperature performance, then aging degree, and last selected test temperature in γ -time curves results; R , J_{nr} , and G_v values results indicate aging degree has a significant effect on binders, and improving binders' aging performance can greatly improve the high-temperature performance.

Keywords: High-temperature performance, waste-based high-viscosity asphalt binders (WHABs), TMA, Repeated Creep Recovery (RCR) test, aging.

31 **Introduction**

32 As one of the most important properties for asphalt pavement, high-temperature
33 performance reflects the ability of asphalt pavement to withstand long-term vehicle
34 loads and pavement diseases like rut-resistance in high temperature environment
35 during service life. Therefore, the high-temperature performance is highlighted in the
36 design and construction specifications as a mandatory requirement (Witczak et al.
37 2002; AASHTO TP63 2007; AASHTO T324 2013). Generally, there are mainly two
38 factors affecting the high-temperature performance of asphalt mixture, i.e., suitable
39 gradation and asphalt binder with excellent high-temperature performance (Sousa et
40 al. 1991; Moghaddam et al. 2011). And once the gradation is fixed, the
41 high-temperature performance of asphalt binder directly determines that of the asphalt
42 mixture (Petersen et al. 1994). It is of great significance to study the high-temperature
43 performance of asphalt binder for better decrease high-temperature diseases in
44 pavement and extend service life (Akisetty et al. 2009; Wang et al. 2012).

45 For decades, the research on experimental methods and evaluation indexes of
46 asphalt binder high-temperature performance does not like that of asphalt mixtures
47 (such as rutting), receiving enough attention, and hence causes a relatively slow
48 development. In the early study, penetration and softening point are considered to be
49 effective indicators to evaluate the temperature susceptibility and high-temperature
50 performance of asphalt binder (Abdellatif et al. 1996; Martins et al. 2013). It is
51 considered that the lower penetration value and higher softening point value, the
52 better high-temperature performance of asphalt binder (AASHTO T49 2015; ASTM

53 D36M 2012; Yetkin. 2007). These two indicators have a good positive correlation
54 with the high-temperature performance of the neat asphalt binder. However, with the
55 widely use of various modified asphalt binders, they have been proven that there are
56 some limitations on correctly reflecting the high-temperature performance of asphalt
57 binders (Shu et al. 2011; Zoorob et al. 2012; Yu et al. 2007). For example, the asphalt
58 binder modified by nano silica and pretreated rubber, could have very close
59 penetration and soften point values to SBS modified asphalt binder, while actually
60 have quiet different viscoelastic behavior (Han et al 2017). Viscosity reflects the
61 fluid's resistance to motion and has been used to evaluate the comprehensive
62 performance of asphalt binder for long (Akisetty et al. 2009; Hesami et al. 2012; Li et
63 al. 2016). Experiments methods such as Brookfield rotational viscometer, kinematic
64 viscosity, and vacuum capillary viscometer have been developed to evaluate the
65 apparent viscosity, kinematic viscosity, and dynamic viscosity of asphalt binder
66 respectively, and the results can reflect the high-temperature performance of asphalt
67 binder indirectly (Akisetty et al. 2009). With the advent of high-precision instruments,
68 dynamic shear rheometer and related rheological viscosity indicators like Zero Shear
69 Viscosity (ZSV) and Low Shear Viscosity (LSV) are also applied to evaluate the
70 high-temperature performance of asphalt binder (Zoorob et al. 2012; Biro et al. 2009;
71 Anderson et al. 2002; Morea et al. 2010; Morea et al. 2011). Theoretically, the
72 above-mentioned viscosity indexes, on the basis of flow resistance of asphalt binder,
73 could be positively correlated with the high-temperature performance of asphalt
74 binder, and hence can be employed to reflect the high-temperature performance of

75 asphalt binder. However, viscosity indexes also exist defects in distinguishing and
76 predicting the high-temperature performance of some high-viscosity asphalt binders
77 like Lake asphalt binder, nano-clay modified asphalt binder and rubber asphalt binder
78 exactly (Stastna et al. 2003; Zaniewski et al. 2004; Widyatmoko et al. 2005).
79 Moreover, in practical application, these viscosity indicators have the disadvantages
80 of time-consuming and complex computation process (Morea et al. 2011; Zoorob et
81 al. 2012). Besides, the test methods of Brookfield viscosity, kinematic viscosity, and
82 vacuum capillary viscometer are greatly subjected to temperature limitations, and
83 would have relatively large test error under improper test temperature or for some
84 high-viscosity asphalt binders. SHRP program recommends $G^*/\sin \delta$ as the indicator
85 to evaluate the high-temperature performance of asphalt binder. However, with the
86 in-depth research on $G^*/\sin \delta$, it has been found that $G^*/\sin \delta$ is not strongly related to
87 the anti-rutting performance of asphalt mixtures and asphalt pavements (Zoorob et al.
88 2012; Bahia et al. 2001; D'Angelo 2009). Therefore, $G^*/\sin \delta$ cannot be taken for an
89 effective evaluation index for high-temperature performance of asphalt binder. In
90 recent years, Repeated Creep Recovery (RCR) test and Multiple Stress Creep
91 Recovery (MSCR) test are considered to be more accurate methods (Delgadillo et al.
92 2006; D'Angelo et al. 2007; Masad et al. 2009; Eric et al. 2014). Unlike the
93 controlled-strain mode by general rheological tests, RCR and MSCR tests both adopt
94 controlled-stress mode, and perform a test cycle consisting 1s of loading and 9s of
95 unloading to detect the recovery and residual shear strains of tested sample (Fig.1).
96 The differences of two tests lies in that RCR test conducts a constant shear stress

(such as 0.1KPa) for 100 repeated cycles, while MSCR test uses two shear stresses (0.1KPa and 3.2KPa) for 10 cycles respectively. In terms of high-viscosity asphalt binder, which has high flow resistance and good deformation recovery, and can not easily reach fatigue point in small number of cycles, seems more likely to adopt RCR test to evaluate its high-temperature performance compared to MSCR test. Table 1 shows the accuracy, advantages, and limitations of above mentioned evaluation indicators for high-temperature performance of asphalt binder.

In this study, four different high-viscosity asphalt binders (three WHABs, including D-I, D-II and F, and a control samples TAFPACK-SUPER modified asphalt binder, TMA) were selected to evaluate their high-temperature performance. According to the above literature review, RCR test is considered as the primary test method. Meanwhile, considering the advantages of MSCR test, this study also set different shear stresses (50Pa, 100Pa, 200Pa, 300Pa) to investigate the effect of shear stress on high-temperature performance. Additionally, the different test temperatures (64°C, 70°C, 76°C) and aging degrees of binder (unaged, TFOT 5h, TFOT 10h) were also studied, and their evaluation indexes (R , J_{nr} , and G_v) were also compared and discussed.

Binders preparation and experimental methods

Binders preparation

In this study, raw materials include SK-90 neat asphalt binder, 30-80 mesh waste rubber powder, waste lubricating oil, and some additives including aging agent, compatibilizer, and tackifier. Based on these, three waste-based high-viscosity asphalt

binders (WHABs, including D-I, D-II and F) were prepared. Meanwhile, TAFPACK-SUPER Modified Asphalt (TMA), which is widely used and highly recognized in China, were selected as a control sample. The details of above mentioned materials and prepared binders can be found in a submitted study^a, which were clearly displayed in *Section 2.1 Raw materials and binders preparation*.

Experimental methods and evaluation indexes

Experiment methods

As mentioned before, RCR test with variable shear stress is conducted to evaluate the high-temperature performance of four binders (D-I, D-II, F and TMA). At selected test temperature and shear stress, the tested sample was subjected to 100 cycles of repeated shear creep recovery based on the dynamic shear rheometer, and each cycle consists of 1s of loading and 9s of unloading (Rodrigo et al. 2006a; D'Angelo et al. 2006). Taking the service temperature and conditions of asphalt pavement in China into account, the test temperatures were set as 64°C, 70°C and 76°C, and the shear stresses were set as 50Pa, 100Pa, 200Pa and 300Pa. Moreover, in order to study the aging effect on the high-temperature performance, the tested binders after Thin Film Oven Test (TFOT) 5h and 10h were also conducted RCR test under different shear stresses at 70°C.

Evaluation indexes

First, after RCR test, γ -time curves showing shear strain (γ) of four binders' changes with cycles (time) under different test conditions were obtained and the

^a Jun Cai, Chen Song, Bochao Zhou et al., titled "Investigation on high-viscosity asphalt binder for permeable asphalt concrete with waste materials", submitted to Journal of Cleaner Production, manuscript number: JCLEPRO-S-18- 08084 (under review).

sample deformation could be reflected. Second, the recovery compliance (R), non-recoverable creep compliance (J_{nr}), and viscous stiffness modulus (G_v) under different test conditions were further calculated to reflect performance changes in RCR tests. G_v is the reciprocal of the creep compliance of the asphalt binder and is the average of fitted values of G_v of the 50th and 51th cycles in RCR test (Rodrigo et al. 2006b; Tan et al. 2010). The calculation formulae of R , J_{nr} , and G_v are shown as Equation (1)-(4), respectively.

$$(1) \quad R = \frac{\gamma_p - \gamma_{nr}}{\gamma_p - \gamma_0}$$

$$(2) \quad J_{nr} = \frac{\gamma_{nr} - \gamma_0}{\tau}$$

Where:

γ_p : peak shear strain per load cycle;

γ_{nr} : residual shear strain per load cycle;

γ_0 : initial shear strain per load cycle.

$$(3) \quad J_t = J_0 + J_1 \left[1 - \exp\left(-\frac{t}{\eta_1 J_1}\right) \right] + J_v$$

$$(4) \quad G_v = \frac{1}{J_v}$$

Where:

J_v : creep compliance of asphalt binder;

J_0 : transient or glassy shear compliance,

J_1 : delay compliance;

η : viscosity of Kelvin model (Pa.s).

Based on the description in Sections *Experimental methods and evaluation indexes*, the details of this study can be concluded in Table 2.

Result and discussion

γ -time curves under RCR test

Fig.2 shows the γ -time curves of four binders under different shear stresses and temperatures. As can be seen from the figure, firstly, the shear strain of each asphalt binder increased gradually with the increasing load cycles. The shear strain of TMA was obviously greater than those of WHABs at different shear stresses and temperatures, indicating the temperature stability of TMA is the worst among the four. Secondly, it can be observed that the rise of test temperature had different influences on the four binders with different shear stresses in terms of γ growth rate as the evaluation index. When the shear stress was relatively small (50Pa), the rise of test temperature had limited influence on the growth of shear strain. However, when the shear stress reached to 200Pa and 300Pa, the rise of test temperature significantly accelerated the deformation of four binders. Additionally, Fig.2 also reveals that shear stress seemed to have more significant influence on the shear strain. It significantly increased the shear strain of four binders at the selected test temperature, and resulted in deterioration of the binders' high-temperature stability. In summary, TMA has the worst high-temperature stability among four binders, and in the RCR test, the shear stress has a more pronounced effect on the high-temperature performance of the binders than the test temperature.

Fig.3 shows the γ -time curves of four binders with different stress at 70°C after

TFOT 5h. It can be seen that TMA had the largest shear strain under each shear stress, and it is noteworthy that the shear strain of D-II also exhibited a significant increase, indicating the short-term aging resistance of D-II is not as good as those of D-I and F. Therefore, after a short-term aging, the high-temperature performance of four binders from good to bad is D-I and F (tied for the best), D-II, and TMA.

Fig.4 shows the γ -time curves of four binders with different stress at 70°C after TFOT 10h. From the figure, it can be seen that after TFOT 10h, the shear strain of TMA was far greater than those of WHABs (D-I, D-II, and F), indicating that the aging resistance of TMA is the worst among the four. In order to more clearly present the changes of D-I, D-II and F, the γ -time curve of TMA at 200 Pa and 300 Pa was omitted. From the curves of D-I, D-II and F, it can be seen that shear strain curves of the three are similar, indicating that the three binders have relatively similar shear stress sensitivity at 70°C after TFOT 10h. The reason may be that D-II after TFOT 5h has no obvious change in high-temperature performance during continued aging treatment. This means after the short-term aging, the aging process of D-II is slower, probably because the internal plasticizer changes into tackifiers, and causes a stronger effect on aging resistance than those of D-I and F during the residual aging process. Therefore, after 10h of TFOT, the high-temperature performance of TMA is the worst, while the high-temperature performance of D-I, D-II and F are similar.

In addition, by comparing the shear strains of four binders under different aging conditions at 70°C in Fig.2-4, it can be seen that the effect of aging degree on the strain in Fig.2-4 is larger than that of the test temperature in Fig.2 based on the

increasing rate of strain, thus it can be considered that the aging degree of the asphalt binder is also an important factor affecting its high-temperature performance.

Based on above analyses, D-I and F have the best high-temperature performance, followed by D-II, and TMA the worst. In terms of the effects of selected parameters on binders in RCR test, shear stress is the most important influencing factor, followed by the aging degree; while test temperatures set in the test exhibit smaller influence except when the shear stress is large.

R, J_{nr} and G_v results in RCR test

R, J_{nr} and G_v results under different temperatures and shear stresses

Fig.5 is the R values of four binders under different temperatures and shear stresses. First, in general, R values of four binders are F, D-I, D-II, and TMA from high to low. R values of WHABs are similar and significantly higher than that of TMA, indicating the recovery ability of TMA is significantly worse than those of WHABs. Second, as the shear stress increases, R values of four binders show a decreasing trend, and R values of TMA and D-II change more obvious. For example, when the shear stress increased from 50 Pa to 300 Pa at 76°C, the R-values of D-II and TMA decreased from 0.96 and 0.90 to 0.84 and 0.76, respectively, which is decreased by 12.5% and 15.6%, respectively; while D-I and F were only decreased by 2.1% and 6.25%, respectively. This reveals that the R values of TMA and D-II are more sensitive to changes of shear stress. Third, as the test temperature rises, R values of four binders also show different changes. When the shear stress was relatively small (50Pa), the temperature change had little effect on R values of four binders, but when the shear

stress increased, TMA and D-II showed greater change in R values than F and D-I, indicating that D-II and TMA are more sensitive to temperature changes under relatively large shear stress. Both D-II and TMA are more sensitive to shear stress and temperature change than F and D-I, so they represent greater changes in R value and poorer recovery ability.

Fig.6 shows the J_{nr} values of four binders at different test temperatures under different shear stresses. From the figure, we can know that first, the order of J_{nr} values of four binders is almost the same, that is, TMA largest, D-II later, then D-I, and last F. Meanwhile, J_{nr} values of WHABs are similar and much smaller than those of TMA, indicating that TMA has the larger viscous content in RCR test. Second, as the shear stress increases, the J_{nr} values of four binders increase, and the increasing ratio of TMA is much more than those of WHABs, indicating that TMA is more sensitive to shear stress changes. Third, as the test temperature rises, the J_{nr} values of four binders are increasing, and the increase in TMA is much greater than those of WHABs. In summary, the J_{nr} values indicate that TMA is more sensitive to shear stress and test temperature, and has larger J_{nr} values for its larger viscous contents.

Fig.7 shows the G_v values of four binders at different test temperatures and shear stresses. It can be known from Fig.7 that, first, the order of G_v values for four binders is almost the same at different shear stresses and test temperatures, i.e., F is the largest, later D-I, then D-II, and last TMA, indicating F has the strongest resistance to non-recoverable deformation (caused by viscous component in binder) and the best high-temperature stability, followed by D-I, D-II, and TMA. Secondly, as the

temperature increases, G_v values of four binders gradually decrease, indicating that four binders are more likely to undergo non-recoverable deformation at higher temperatures, i.e., their high-temperature stability is weakening at higher temperatures. Based on the above analysis, it can be seen that TMA has the worst resistance to non-recoverable deformation and the worst temperature stability among four binders.

R, J_{nr} and G_v results under different aging degree and shear stresses

Fig.8 shows the R values of four binders under different aging degree and shear stresses. Several results can be drawn from the figure. First, the R values of WHABs are similar and are significantly greater than those of TMA, suggesting the high temperature recovery capabilities of WHABs are significantly better than those of TMA. Second, with different shear stresses, the R values of four binders does not change significantly, indicating that the shear stress is not a major factor at 70°C. Third, the R values of TMA after aging is significantly reduced and show a cliff-fall after TFOT 10h. The effect of aging on TMA is much greater than those on WHABs, which indicates that the aging resistance of TMA is significantly lower than those of WHABs.

Fig.9 shows the J_{nr} values of four binders under different aging degree and shear stresses. It can be known from the figure that first, with different shear stress, the J_{nr} value of TMA is significantly higher than those of WHABs, indicating that the TMA has the most viscous components and the worst high-temperature performance. Second, when the shear stress is small (50Pa, 100Pa), the J_{nr} values of WHABs are

similar, but when the shear stress is larger (200, 300Pa), the J_{nr} values of D-II are higher than those of F and D-I. D-II is more sensitive to larger shear stress, and its high-temperature performance is worse than those of F and D-I. Third, aging has a greater impact on both TMA and D-II. The sharp increase in the J_{nr} value of TMA as the aging deepens indicates that TMA has a strong sensitivity to aging, which is consistent with the results obtained from R value of TMA. After TFOT 5h, the J_{nr} value of D-II increased by a large margin while decreased slightly after TFOT 10h, which may be caused by aging resistance of more internal tackifiers and is consistent with the conclusion got from γ -time curve. In summary, the J_{nr} value of TMA is larger than that of WHABs, and TMA is more sensitive to aging and has poorer aging resistance.

Fig.10 shows the G_v values of four binders under different aging degree and shear stresses. We can know that first, the G_v values of D-I and F are the highest and are similar to each other, followed by D-II and TMA, indicating that D-I and F have the largest resistance to non-recoverable deformation and TMA has the worst. Second, as the degree of aging deepens, the G_v values of four binders continue to decrease and the decrease rates of D-I and F are even larger than that of TMA, but their G_v values are still much higher than that of TMA. This shows that aging has a significant impact on the resistance to non-recoverable deformation of four binders.

According to the above analyses, some conclusions can be drawn. First, all three evaluation indexes reflect that TMA has the worst high-temperature performance, followed by D-II, and D-I and F are the best, which is consistent with the results

obtained by the γ -time curve. Second, it is worth noting that R , J_{nr} , and G_v values all indicate that aging has a significant effect on each binder, especially for R and G_v , where aging is even more influential than shear stress. Therefore, it can be considered that it is of important significance to improve the aging resistance property of the binder so as to improve the high-temperature performance of the binder.

G_v results under different shear stresses

As mentioned in Section 2.2.2, G_v , as an evaluation index reflecting the non-recoverable modulus in the Burgers model and embodying the resistance to non-recoverable deformation, is a constant value independent of shear stress and shear strain (Masad et al. 2009). Fig.11 and Fig.12 are G_v values of four binders with different shear stresses and at different temperatures and different aging degree. From Fig.11, it can be seen that the G_v values of all binders are more unstable overall at lower test temperatures, which reflects the inaccuracy of G_v in terms of evaluating high-temperature performance of high-viscosity asphalt binder. The main reason is that during the unloading time (9s) in RCR test, the residual deformation of each binder may not be completely eliminated, resulting in different residual deformation of different binders, and this residual deformation is recovered slower at lower temperatures, so G_v values are less stable at low temperatures. Fig.12 shows that the G_v values of different binders decrease sharply with the aging deepening, indicating that aging has a strong influence on the G_v value of each binder.

Conclusion

In this study, the test methods and evaluation indexes for high-temperature

performance of asphalt binder were reviewed and compared, then RCR test with various shear stresses was conducted to test high-temperature performance of four high-viscosity asphalt binders (TMA, D-I, D-II and F), and the γ -time curve, R values, J_{nr} values and G_v values were used to further evaluate the test results. The following conclusions were obtained:

- Different asphalt binders require different test methods and evaluation indexes to evaluate their high-temperature performance, and for high-viscosity asphalt binders, RCR test with various shear stresses is more appropriate.
- Based on the analyses of γ -time curve, R values, J_{nr} values and G_v values, the high-temperature performance of four asphalt binders from good to bad is F, D-I, D-II and TMA.
- The γ -time curve shows that among the three factors, i.e., test temperature, shear stress and aging degree of binders, the shear stress has the most obvious effect, followed by aging degree, and the test temperature the least.
- R, J_{nr} , and G_v values all indicate that aging degree has a significant effect on the high-temperature performance of all binders. It is of important significance to improve the aging resistance property of the binder so as to improve the high-temperature performance of the binder.
- As an evaluation indicator for the high-temperature performance of asphalt binders, G_v value has limitations in accurately reflecting the viscous properties of asphalt binders, especially in low temperatures.

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Table 1 Comparison of evaluation indicators for high-temperature performance of asphalt binder

Evaluation Indicators	Penetration and softening point	Viscosity		G*/sin δ	RCR	MSCR
		Brookfield rotational viscometer, kinematic viscosity, and vacuum capillary viscometer	ZSV, LSV			
Correlation with high-temperature performance	Low	Middle	High	Low	High	High
Advantages	Simple equipment, easy operation, high correlation with neat asphalt	Reflect the high- temperature performance of most asphalt binder at a relative high accuracy		easy operation,	high correlation with high-temperature performance	easy operation, high correlation with most asphalt binder
Limitations	Not suitable for evaluating the high-temperature performance of modified asphalt	Complex operation, time-consuming, temperature limitation , uncontrollable errors and not suitable for high-viscosity asphalt binders	Complicated calculation, time-consuming, not suitable for high-viscosity asphalt binders	Low correlation with high-temperature performance	Time-consuming	Not suitable for high-viscosity asphalt binders
References	(Shu et al. 2011; Zoorob et al. 2012; Yu et al. 2007)	(Zoorob et al. 2012; Morea et al. 2011; Widyatmoko et al. 2005; Zaniewski et al. 2004; Stastna et al. 2003)		(Zoorob et al. 2012; Bahia et al. 2001; D'Angelo 2009)	(Rodrigo et al. 2006a; D'Angelo et al. 2007; Masad et al. 2009; Eric et al. 2014)	

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Table 2 The details of RCR test conditions for high-viscosity asphalt binders

RCR test	Binders	Aging degree	Test temperature	Shear stress	Evaluation indexes
100 cycles, 1s of loading and 9s of unloading in each cycle.	D-I, D-II, F and TMA	Unaged, TFOT 5h and TFOT 10h	64°C, 70°C and 76°C	50Pa, 100Pa, 200Pa and 300Pa	R, J_{nr} , and G_v

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Fig.2: RCR images of unaged samples with different shear stresses and temperatures

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b) 50 Pa and 70°C

c) 50 Pa and 76°C

d) 100 Pa and 64°C

e) 100 Pa and 70°C

f) 100 Pa and 76°C

g) 200 Pa and 64°C

h) 200 Pa and 70°C

i) 200 Pa and 76°C

j) 300 Pa and 64°C

k) 300 Pa and 70°C

l) 300 Pa and 76°C

Fig.3: RCR images of TFOT 5h samples with different shear stresses at 70°C

a) 50 Pa

b) 100 Pa

c) 200 Pa,

d) 300 Pa

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488 Fig.4: RCR images of TFOT 10h samples with different shear stresses at 70°C

489 a) 50 Pa

490 b) 100 Pa

491 c) 200 Pa

492 d) 300 Pa

493

494 Fig.5 R values of four samples in different temperatures and shear stresses

495 a) 50 Pa

496 b) 100 Pa

497 c) 200 Pa

498 d) 300 Pa

499

500 Fig.6: J_{nr} results of four samples in different temperatures and shear stresses

501 a) 50 Pa

502 b) 100 Pa

503 c) 200 Pa

504 d) 300 Pa

505

506 Fig.7: G_v results of four samples in different temperatures and shear stresses

507 a) 50 Pa

508 b) 100 Pa

509 c) 200 Pa

510 d) 300 Pa

511

512 Fig.8: R results of four samples in different aging degree and shear stresses

513 a) 50 Pa

514 b) 100 Pa

515 c) 200 Pa

516 d) 300 Pa

517

518 Fig.9: J_{nr} results of four samples in different aging degree and shear stresses

519 a) 50 Pa

520 b) 100 Pa

521 c) 200 Pa

522 d) 300 Pa

523

524 Fig.10: G_v values of four samples in different aging degree and shear stresses

525 a) 50 Pa

526 b) 100 Pa

527 c) 200 Pa

528 d) 300 Pa

529

530 Fig.11: G_v values of four samples in different temperatures and shear stresses

531 a) TMA

532 b) D- I

533 c) D- II

534 d) F

535

536 Fig.12: G_v values of four samples in different aging degree and shear stresses

537 a) TMA

538 b) D- I

539 c) D- II

540 d) F

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542 **Fig.1: Details of the relationship between shear strain and test time in one cycle of RCR test**

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545 **Fig.2 RCR images of unaged samples with different shear stresses and temperatures: a) 50**546 **Pa and 64°C, b) 50 Pa and 70°C, c) 50 Pa and 76°C, d) 100 Pa and 64°C, e) 100 Pa and 70°C,**547 **f) 100 Pa and 76°C, g) 200 Pa and 64°C, h) 200 Pa and 70°C, i) 200 Pa and 76°C, j) 300 Pa**548 **and 64°C, k) 300 Pa and 70°C, l) 300 Pa and 76°C**

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577 **Fig.12 G_v values of four samples in different aging degree and shear stresses: a) TMA, b)**578 **D-I, c) D-II, d) F**

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