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Investigating the High- and Low-temperature Performance of Warm Crumb Rubber Modified Bituminous Binders using Rheological Tests

Haopeng Wang, Ph.D. candidate

Section of Pavement Engineering, Faculty of Civil Engineering & Geosciences, Delft University of Technology, Stevinweg 1, 2628 CN, Delft, The Netherlands;

Department of Civil and Environmental Engineering, The Hong Kong Polytechnic University, Hong Kong, China.

Email: haopeng.wang@tudelft.nl

Xueyan Liu, Ph.D., Associate professor

Section of Pavement Engineering, Faculty of Civil Engineering & Geosciences, Delft University of Technology, Stevinweg 1, 2628 CN, Delft, The Netherlands

Panos Apostolidis, Ph.D. candidate

Section of Pavement Engineering, Faculty of Civil Engineering & Geosciences, Delft University of Technology, Stevinweg 1, 2628 CN, The Netherlands

Di Wang, Postdoctoral researcher

Department of Civil Engineering - ISBS, Technical University of Braunschweig, Braunschweig, Germany

Zhen Leng, Ph.D., Associate professor

Department of Civil and Environmental Engineering, The Hong Kong Polytechnic University, Hong Kong, China

Guoyang Lu, Ph.D., Research assistant professor (Corresponding author)

Department of Civil and Environmental Engineering, The Hong Kong Polytechnic University, Hong Kong, China

Sandra Erkens, Ph.D., Professor

Section of Pavement Engineering, Faculty of Civil Engineering & Geosciences, Delft University of Technology, Stevinweg 1, 2628 CN, The Netherlands

Athanasios Sarpas, Ph.D., Professor

Department of Civil Infrastructure and Environmental Engineering, Khalifa University, Abu Dhabi, United Arab Emirates;

Section of Pavement Engineering, Faculty of Civil Engineering & Geosciences, Delft University of Technology, Stevinweg 1, 2628 CN, The Netherlands

ABSTRACT

Rubberized asphaltic materials have been frequently combined with warm mix asphalt technologies to tackle the issues of high energy consumptions and emissions during construction. Effective and accurate characterization of binder properties is conducive to the improvement of long-term pavement performance. The current study aims to quantify the effects of rubber content and warm-mix additives on rutting and thermal cracking performance of crumb rubber modified bitumen (CRMB), and explore the rubber and additives modification mechanisms and their impacts on the binder performance. CRMBs containing different rubber contents and warm-mix additives after long-term aging were subject to multiple stress creep and recovery (MSCR) tests and low-temperature frequency sweep tests using a dynamic shear rheometer (DSR) with the 4-mm loading plate, to investigate the high- and low-temperature performance, respectively. Rheological tests were also conducted on the bitumen and rubber phases of CRMB to understand the rubber modification mechanism. Results show that CRMB binders have superior rutting and thermal cracking resistance due to rubber modification. The improvement of high- or low-temperature performance is more prominent at higher rubber concentrations. The effects of warm-mix additives on the rutting and thermal cracking performance are different. Generally, the wax-based additive improves the rutting resistance but negatively affects the low-temperature performance. By contrast, the chemical-based additive has an opposite effect except for the high-temperature performance of neat bitumen. The stiffening of the bitumen phase and the contribution of swollen rubber particles in the bitumen matrix together contribute to the peculiar viscoelastic response of CRMB, i.e., stiffer/softer and more elastic at high/low temperatures. This modification mechanism explains the superior rutting and thermal cracking performance of CRMB.

Keywords: Bitumen, Crumb rubber, Warm-mix additives, Rutting, Thermal cracking, Rheology

INTRODUCTION

Crumb Rubber Modified Bitumen

Bitumen modification using crumb rubber from scrap tires has been increasingly popular in the context of circular economy (Leng et al. 2018; Nanjegowda and Biligiri 2020). Although crumb rubber modified bitumen (CRMB) has been reported to improve the engineering properties of binders (Singh et al. 2017; Wang et al. 2018), its high viscosity has caused some concerns during construction, such as poor pumpability, mixability, and workability (Wang et al. 2018; Yu et al. 2018). Besides, the requirement of higher production temperatures of rubberized asphalt mixtures increases the heat energy consumption and greenhouse gas emissions and generates more asphalt fumes, which compromises the working conditions of paving crews at the construction site (Farshidi et al. 2013). The emergence of warm mix asphalt (WMA) technologies provides an effective solution to the above issues by decreasing the construction temperatures of rubberized asphalt (Wang et al. 2018; Yu et al. 2017). Since the combination of CRMB with WMA is a relatively new implementation in the sustainable asphalt paving industry, it is important to elucidate its long-term performance characteristics either in the laboratory or in the field.

Permanent deformation, fatigue, and thermal cracking are the primary distress modes of asphalt pavements. Past research has demonstrated that the performance-related properties of the bituminous phase has predominant effects on the durability of asphalt mixture (Bahia et al. 2001). The effective and accurate characterization of binder performance is of great importance from the point of view of its potential applications (Hajj and Bhasin 2017): (a) as a binder grading tool for design specification; (b) as a screening/ranking tool to examine the influence of modifiers and additives; (c) to obtain fundamental material properties of binders to be used as model inputs to predict the mechanical performance of bituminous mixtures. Past studies have looked into the effects of rubber modification and various WMA additives on the rutting and thermal cracking performance of CRMB binders through traditional test methods and parameters (Daly and Negulescu 1997; Kim et al. 2010; Sebaaly et al. 2003; Singh et al. 2017). It was generally reported that rubber modification can improve the binders' resistance to permanent

deformation after long-term aging. Although rubber modification has been seen to improve the low-temperature performance of binders, cautions need to be taken to properly design the CRMB, e.g., size and content of crumb rubber (Sebaaly et al. 2003). In terms of WMA additives, their effects on the properties of CRMB vary with the type of used WMA additives. Organic additives and chemical additives usually affect the rutting or thermal cracking performance of CRMB binders in a different manner (Akisetty et al. 2009; Kim et al. 2014; Yu et al. 2016). Therefore, it is necessary to characterize the high- and low-temperature performance of binders for specific material combinations.

The fatigue performance of warm CRMB has been systematically investigated in a previous study (Wang et al. 2020). Usually, contradictory measures were taken to increase the resistance of binders to permanent deformation and thermal cracking. Balancing the high- and low-temperature performance is a major challenge to mitigate these distresses. The current study emphasizes characterizing high- and low-temperature performance and exploring the mechanism of the rubber modification to optimize the binder design and to extend the durability of rubberized asphalt pavements.

Binder Characterization for Rutting

Under repeated traffic loadings, the accumulated strain in the bituminous binder is regarded as the main reason for the rutting of asphalt pavements. Different methods and criteria have been developed to characterize the propensity of a binder to permanent deformation under periodic loading. The Superpave performance grading (PG) system uses the viscoelastic parameter ($G^*/\sin \delta$) measured from DSR as a rutting parameter at high temperatures. The rutting parameter obtained in the linear viscoelastic range can estimate the total dissipated energy during a loading cycle, which is believed to be related to rutting. To minimize rutting, the energy dissipated per loading cycle should be minimized. Hence in a stress-controlled test, the rutting parameter $G^*/\sin \delta$ should be maximized for binders at both fresh and short-term aged states (Anderson and Kennedy 1993). However, many studies have demonstrated the inadequacy of $G^*/\sin \delta$ in correlating with mixture rutting performance, especially for polymer modified binders (Bahia et al. 2001). Attempts have been made to replace this inaccurate rutting parameter.

Sybilski (1994) proposed the concept of the zero-shear viscosity (ZSV) at 60 °C to characterize the binder's rutting resistance. Good correlations between ZSV of binders and rutting performance of mixtures were observed. However, other researchers found that highly polymer-modified binders, which behave as viscoelastic solids, exhibit extremely high viscosity gradients at low shear rates, leading to unrealistically high ZSV values using model predictions (De Visscher and Vanelstraete 2004). Alternatively, the concept of low shear viscosity (LSV) was introduced to overcome this issue (Morea et al. 2010).

Since the rutting parameter was obtained using small cyclic reversible loadings, the contribution of binder damage accumulation was not considered. To address this, Bahia et al. (2001) developed the repeated creep recovery (RCR) test using DSR to simulate the intermittent nature of traffic loading during the NCHRP 9-10 project. The RCR test can separate the dissipated energy between permanent deformation and delay elasticity. A novel parameter G_v derived from the Burger's model was introduced to characterize the binder's resistance to permanent deformation. Considering the significant stress dependency of polymer modified binders and different loading stress conditions, D'Angelo et al. (2007) improved the RCR by introducing multiple stress creep and recovery (MSCR) tests. The MSCR is a simple and performance-related test for both unmodified and modified binders. The non-recoverable creep compliance (J_{nr}) and percent recovery were used to characterize the stress dependence and elastic response of bituminous binders. Parameters from MSCR tests have been demonstrated to have a much better correlation to mixture rutting performance than the existing Superpave binder criteria (D'Angelo 2009).

Binder Characterization for Thermal Cracking

Thermal cracking, including low-temperature shrinkage cracking and thermal fatigue cracking, is the primary distress of asphalt pavements operating in cold regions such as northern American and northern Europe. In Europe, most of the thermal cracking is low-temperature shrinkage cracking. With the decrease of temperature, significant tensile stresses will develop in the asphalt surface layer and ultimately lead to

the initiation and propagation of cracks. As cracks mainly occur in the binder or mastic phase, it is vital to select appropriate binders in mix design that satisfy the requirements of low-temperature performance (Hajj et al. 2019). Many methods were proposed to evaluate the low-temperature properties of binders, such as the Fraass breaking point test and low-temperature ductility test. However, these traditional tests cannot accurately predict the critical temperature at which thermal cracking occurs (Wang et al. 2017). The inadequacy is more significant when using these tests for the assessment of polymer modified binders (Isacsson and Lu 1995).

The current Superpave PG system utilizes the bending beam rheometer (BBR) test to determine the low-temperature PG of binders. Two parameters (i.e., creep stiffness and m -value (relaxation rate)) obtained from the BBR test were used to determine the critical cracking temperature by applying two threshold limits on them. The low-temperature PG of binders is ruled as the higher of below two values based on two criteria: the temperature above which the maximum creep stiffness should not exceed 300 MPa and the temperature above which the minimum m -value should not be less than 0.3. The creep stiffness is directly related to the thermal stress built up in the bituminous material when temperature declines. Meanwhile, the m -value shows the ability to retain the viscoelasticity and relaxing the accumulated thermal stress.

Standard BBR testing requires a relatively large amount of materials (15 g for each beam sample) which may be inconvenient and difficult when assessing recovered binders or emulsion residues. Besides, BBR tests often underestimate the low-temperature performance of polymer modified bitumen (PMB) (Lu et al. 2017). To overcome this limitation, researchers have explored the possibility of using a DSR with parallel plates of 4 mm in diameter (4-mm DSR) as an alternative to the BBR (Farrar et al. 2015; Lu et al. 2017; Oshone 2018; Sui et al. 2011; Wang et al. 2019). Besides the primary advantage of requiring a small amount of binder, 4-mm DSR is a simple and more accurate method than BBR when measuring the low-temperature rheological properties to determine the accumulated thermal stress (Farrar et al. 2013). It was also reported that 4-mm DSR has good repeatability and good consistency among data collected from different DSR plate sizes (Hajj et al. 2019; Sui et al. 2010).

RESEARCH OBJECTIVES AND SCOPE

The objectives of this study include: (1) to quantify the effects of rubber content and warm-mix additives on rutting and thermal cracking performance of CRMB binders through new-generation rheological tests; and (2) to explore the modification mechanism of rubber and additives for the binder performance. MSCR tests were performed on the short-term aged binder samples to characterize the rutting performance. 4-mm DSR frequency sweep (FS) tests were performed on the long-term aged binder samples to characterize the thermal cracking performance. Furthermore, FS tests were also done on the bitumen and rubber phases of CRMB to gain insights into the rubber modification mechanism and its impact on the overall binder performance.

Together with a previous study on fatigue performance (Wang et al. 2020), this study is also part of the attempt to characterize the binder properties in the whole temperature range (high-, intermediate- and low-temperature) with only one DSR instrument using different plate sizes (25 mm, 8 mm, and 4 mm) as shown in Figure 1. This unified DSR method is deemed to be a technical breakthrough enabling improvements in the capacity of providing performance-related specifications for bituminous binders.

MATERIALS AND METHODS

Raw Materials and Binder Sample Preparation

Penetration grade 70/100 bitumen (Nynas) and fine CRMs from scrap truck tires (ambient grounding, size ranging from 0 to 0.5 mm) were used to prepare CRMB binders. High shear mixer was applied to mix the blend of neat bitumen and rubber of different percentages (5%, 10%, 15%, and 22% by weight of neat bitumen) for 30 minutes at 180°C with a mixing speed of 6000 rpm. This mixing procedure was optimized to allow sufficient rubber swelling to achieve optimized rheological properties (Wang et al. 2020). The produced CRMBs were designated as CRMB-5, CRMB-10, CRMB-15, and CRMB-22. Two types of non-foaming warm-mix additives, i.e., wax-based solid additive (W) and chemical-based liquid additive (C), were added to neat bitumen and CRMB-22 at 160°C and mixed for 10 minutes. The resultant binders were designated as 70/100-W, 70/100-C, CRMB-22-W, and CRMB-22-C. The reason why additives were only added to CRMB-22 is that CRMB-22 exhibited extremely high viscosities, which will significantly

influence its pumpability, mixability, and workability if not reduced (Leng et al. 2017). The detailed material properties and binder preparation process can be found in (Wang et al. 2020).

Rotational Viscosity

To examine the effect of rubber modification and warm-mix additives on the binder viscosity, the Brookfield viscometer was used to measure the rotational viscosities of different binders at a constant rotational speed of 20 rpm using the #27 cylindrical spindle (spindle diameter=11.76 mm, side length=33.02 mm, effective length=39.29 mm). Viscosity tests were conducted at various temperatures, 110, 135, 160, 177, and 190°C, which cover the temperature range of production, transport, and construction (Zhang et al. 2015).

Figure 2a shows the rotational viscosities of CRMB binders at various temperatures. It is obvious that binder viscosity decreases with the increase of testing temperature. With the increase of rubber content, the binder viscosity increases dramatically. Even at 190°C, the rotational viscosity of CRMB-22 has exceeded 4000 mPa·s. Excessively high viscosities of CRMB will impede its application in asphalt production and construction. Based on the recommended specification for rubberized bitumen in California (State of California Department of Transportation 2003), the apparent viscosity of CRMB at 190°C should be in the range of 1500-4000 mPa·s to ensure the proper plant production. CRMB binders with rubber contents lower than 15% have acceptable viscosities. Therefore, warm-mix additives need to be incorporated into CRMB-22 to reduce the viscosity. Figure 2b shows the effect of warm-mix additives on the viscosity of CRMB binders. For comparison, additives were also added to neat bitumen. As expected, both additives effectively reduce the viscosity of both neat bitumen and CRMB. Particularly, the viscosity of CRMB-22 has reduced to lower than 4000 mPa·s at 190 °C, which satisfies the specifications for rubberized bitumen as proposed by Caltrans (California Department of Transportation). Therefore, for the rest of the study, warm-mix additives were only added to CRMB-22 and neat bitumen for comparison reason.

Laboratory Ageing Procedure

All fresh binder samples were short-term aged using oven aging as an alternative to the standard rolling thin film oven (RTFO) test. This is because the RTFO ageing procedure encounters flowing and collecting issues for highly viscous binders (e.g., CRMB) (Bahia et al. 1998). A modified oven aging procedure, in which bitumen samples with a thickness of 1.25 mm were aged at 163°C for 2 h, was used to replace the standard RTFO aging procedure in this study (Wang et al. 2020). A part of the short-term aged samples were subject to MSCR tests. The remaining short-term aged samples were further aged by pressure aging vessel (PAV) tests. The long-term aged samples were subject to 4-mm DSR tests.

Preparation of Bitumen and Rubber Phases of CRMB

In terms of bitumen and rubber phases of CRMB, considering the CRMB-22 binder as an example, the bitumen phase of CRMB-22, which is designated as CRMB-22-BP, was extracted by filtering the insoluble rubber particles from CRMB-22 with a mesh sieve (0.063-mm) at 163°C. The swollen rubber samples were prepared to represent the rubber particle phase in CRMB. Cylindrical rubber samples (2-mm in thickness and 8-mm in diameter) cut from waste truck tire tread were soaked in hot bitumen at 180°C to prepare the swollen rubber samples. The detailed sample preparation process illustrated in Figure 3 can be found in (Wang et al. 2020). Both CRMB-22-BP and swollen rubber samples were subject to FS tests by DSR.

MSCR Test Method

The MSCR test was conducted using a DSR with 25-mm parallel plates AASHTO T 350-19. Considering the weather conditions in the Netherlands, the maximum pavement temperature is less than 60°C (Bijsterveld et al. 2001). Therefore, the testing temperature for binder high PG characterization in this study was selected as 64°C, which is regarded as sufficient. The predetermined temperature was stabilized within $\pm 0.1^\circ\text{C}$ tolerance for 10 minutes. The chosen two stress levels (0.1 kPa and 3.2 kPa) can characterize both linear and non-linear responses of binders. The test protocol includes ten creep (1 s) - recovery (9 s) cycles at each stress level. Before the twenty creep-recover cycles for the two stress levels, a first ten cycles at 0.1 kPa were applied for conditioning the sample to reach the steady state to minimize

the effects of delayed elasticity (Golalipour 2011). The non-recoverable creep compliance (J_{nr}) and percent recovery (R) were calculated. Since rutting is primarily critical during the earlier in-service period of pavements at high temperatures, MSCR tests were performed on short-term aged binder samples to simulate the aging after the road construction.

4-mm DSR Test Method

Using 4-mm parallel plates, FS tests from 1 to 100 rad/s were conducted at low temperatures from -30 to 10°C with an incremental step of 10°C. It was reported that DSR measurements at low temperatures may generate errors in the values of dynamic data due to instrument compliance. Using small-diameter plates with a high gap between the plates at low temperatures is recommended to reduce the instrument compliance error (Schroter et al. 2006). In the current study, a gap of 3-mm between the plates was used with the 4-mm parallel plate geometry, which was validated and recommended by other institutions (Wang et al. 2019). Besides, automatic real-time compliance corrections were done using the pre-input machine compliance for the 4-mm parallel plates on the measuring system. To accommodate the specimen on such a small plate, a specifically designed butterfly silicone mold was manufactured to prepare and install the samples on the DSR plates as shown in Figure 4. The detailed sample preparation steps can be found in (Wang et al. 2019). When chamber temperature drops to -30°C, the resulting binder shrinkage could potentially cause its de-bonding from the top plate. To ensure sufficient adhesion between the sample and plates, the normal force was set automatically to zero during the cooling phase. For each type of binder, a minimum of three replicates were tested and average results were reported.

Frequency Sweep Test Method

FS tests of CRMB and bitumen phase binder samples were conducted using the parallel-plate configuration (25-mm diameter and 1-mm gap) from 0.1 to 100 rad/s at temperatures from 10 to 70°C. To ensure measurements within the linear viscoelastic (LVE) range, a strain level of 0.1% under strain-controlled mode was chosen.

Compared to binders, the viscoelastic response of swollen rubber is less sensitive to temperature. Therefore, the rheological properties of swollen rubber were measured using the 8-mm parallel plates over a temperature range of -10~130°C. The measurements were performed at a strain level of 1% under strain-controlled mode from 0.1 to 100 rad/s. The detailed testing setup can be found in (Wang et al. 2020).

RESULTS AND DISCUSSION

Repeatability of DSR Test Results

For modified binders containing particulate matter such as crumb rubber, the variability of binder test results is always a concern for accurate characterization. It was recommended that the gap between the DSR parallel plates should be higher than four times the maximum particulate size (FHWA 2014). This is to eliminate the influence of the particle-to-particle interaction between the parallel plates. Therefore, considering the crumb rubber particle size in this study, the gap height might be accommodated to 2 mm. However, to make comparable results with the conventional DSR setup of 25-mm diameter and 1-mm gap for neat bitumen, the 1-mm gap setting was also applied to the tests of CRMB binders. Test results from three replicates were compared as shown in Figure 5a to verify the validity of the 1-mm gap for CRMB binders. It can be seen that the strain responses of three replicates at different creep-recovery cycles are overlapped with each other. The calculated values of non-recoverable compliance and recovery at 3.2 kPa are also shown in Figure 5a. The coefficients of variation for both $J_{nr,3.2}$ and $R_{3.2}$ are less than 10%, which are generally acceptable for engineering purposes. In terms of the 4-mm DSR test, the variability of FS test results is shown in Figure 5b. Both vertical and horizontal axes are plotted in the arithmetic scale to more clearly see the variance of values. For both complex modulus and phase angle, the three replicates yield quite close results.

To further check the result variability between different replicates, a one-way analysis of variance (ANOVA) was performed on the measured values of complex modulus and phase angle. A confidence level of 95% was used. The ANOVA results are summarized in Table 1. For either shear strain from MSCR test or complex modulus and phase angle from 4-mm DSR FS test, the F value is smaller than the F -critical, which means there is no significant difference between the results from the three replicates. In

addition, the *P-value* is much higher than the significance level, indicating the testing results are quite repeatable and not affected by the rubber particle interactions or sample preparation process. Therefore, the binder tests carried out in this study using the adopted DSR configurations have good repeatability.

High-temperature Rutting Performance

Creep and Recovery Strain Response

Figure 6 shows the strain responses of the first creep and recovery cycle at 3.2 kPa for different binders. A semi-logarithmic plot was used to compare the strain difference more clearly. It can be found from Figure 6a that the accumulated strain of neat bitumen 70/100 increases dramatically under the creep stress. With the increase of rubber content, less accumulated strains of CRMB binders were observed. For instance, the accumulated strain of CRMB-22 after the creep stage was only around 12% of that of CRMB-5. The main creep deformation of neat bitumen is viscous flow at high temperatures while for CRMB binders, it changes to viscoelastic flow due to rubber modification. During the recovery stage, neat bitumen 70/100 almost shows no strain recovery, further indicating that viscous flow is the main type of creep deformation for neat bitumen. It also implies that viscosity can be regarded as a primary parameter to characterize the high-temperature performance of neat bitumen. By contrast, CRMB binders show obvious strain recoveries due to the delayed elasticity. The higher the rubber content, the less non-recoverable strain (permanent deformation). Viscosity is insufficient as a parameter to characterize the high-temperature performance of CRMB binders. Therefore, parameters, which are capable of capturing the delayed elasticity, should be developed to evaluate the rutting resistance of highly modified binders.

Figure 6b shows the effects of two warm-mix additives on the creeping and recovering properties of binders. For the wax-based additive, it decreased the accumulated strain of neat bitumen during the creep stage but did not significantly change the strain recovery ability. By contrast, the addition of wax-based additives maintained the creep strain response but slightly increased the recoverable strain. In either way, wax-additives can facilitate the deformation resistance of binders. For the chemical-based additive, it has an insignificant effect on the creep-recovery response. However, it seemed to soften CRMB-22 by increasing the accumulated creep strain and decreased the recoverable strain, hence reducing the

resistance to permanent deformation. Besides the above findings, the modification of warm-mix additives did not change the main creep deformation mechanism of binders.

Non-recoverable Compliance and Percentage Recovery

Two parameters from MSCR test results, namely non-recoverable compliance and percentage recovery, were used to capture the delayed elasticity and characterized the high-temperature properties of binders. Table 2 summarizes MSCR test results of both neat bitumen and CRMB with warm-mix additives. It is apparent that the J_{nr} values of bitumen at both stress levels significantly decreased as the rubber content increased, indicating an improved rutting resistance of CRMB. In addition, CRMB binders had a considerably higher ability to recover (R) than the unmodified bitumen 70/100 at both stress levels. The lower J_{nr} and higher percent recovery of CRMB compared to neat bitumen resulted from the superior polymer network established by the bitumen-rubber interaction (Wang et al. 2020).

With regard to warm-mix additives, the addition of wax-based additive to both 70/100 and CRMB-22 decreased the J_{nr} value but had an insignificant effect on the percent recovery. The increased resistance to permanent deformation is attributed to the microcrystalline structure of the wax-based additive when temperature drops below its melting temperature (around 110 °C). Uniformly distributed wax particles will form lattice structures at service temperature (64 °C in this case) to stiffen the binder (Yu et al. 2016). The chemical-based additive generally had insignificant effects on the MSCR results of neat bitumen. By contrast, the chemical-based additive seemed to soften CRMB-22 and slightly decreased the percent recovery.

The stress sensitivity parameter $J_{nr diff}$ seems to lack ubiquity to characterize the stress sensitivity of CRMB binders. From Table 2, all CRMB binders except CRMB-5 and 70/100-W exceeded the maximum allowable $J_{nr diff}$ value (75%) as stated in the specification AASHTO M 332-19. However, the low J_{nr} values of these binders demonstrate that they have adequate resistance to permanent deformation. The parameter $J_{nr diff}$ unfairly penalizes binders with low J_{nr} values. Alternatively, the most recent specification AASHTO M 332-19 addresses the concern by removing the $J_{nr diff}$ requirement for binders

having a $J_{nr3.2}$ value of 0.5 kPa^{-1} or lower at the selected test temperature. To remedy the unsuitability of the $J_{nr diff}$ parameter for accreditation purposes, an alternate parameter $J_{nr slope}$ was proposed in Equation 1. It is defined as the change in J_{nr} for an incremental change in applied stress τ (Stempihar et al. 2018).

$$J_{nr slope} = \frac{dJ_{nr}}{d\tau} = \frac{J_{nr3.2} - J_{nr0.1}}{\tau_{3.2} - \tau_{0.1}} \times 100 \quad (1)$$

Where $\tau_{3.2}$ and $\tau_{0.1}$ are the stress levels of 3.2 kPa and 0.1 kPa. $J_{nr3.2}$ and $J_{nr0.1}$ are the corresponding J_{nr} values at the two stress levels. From Table 2, it can be seen that all binders meet the requirement of stress sensitivity (lower than 75%) using the $J_{nr slope}$ parameter, which can be considered as a more appropriate representation of stress sensitivity. $J_{nr slope}$ was also reported to have a much better correlation with an incremental increase in rut depth (Stempihar et al. 2018). CRMB binders exhibited less stress sensitivity as expected using the new parameter. The acceptable $J_{nr3.2}$ and percent differences for varying levels of traffic are specified in AASHTO MP 19-10. Using the alternate parameter $J_{nr slope}$, the traffic level each binder can reach at the temperature of 64°C was also shown in Table 2. Neat bitumen with and without warm-mix additives are suitable for pavements with standard traffic conditions. With the increase of rubber content, CRMB can be used for heavier traffic conditions.

Low-temperature Rheology and Performance Grading

Correlation between 4-mm DSR Measured Data and BBR Parameters

While BBR is a flexural test in the time domain, DSR measures the shear rheological properties of binders in the time/frequency domain. Therefore, FS measurements from DSR must be mathematically transformed to be comparable to those from BBR. Previous studies have developed methods to convert DSR data into the parameters related to BBR tests (i.e., interconversion from dynamic frequency sweep to shear stress relaxation) as shown in Table 3. The corresponding low-temperature PG criteria for binders were also proposed using 4-mm DSR, and they are summarized in Table 3.

Regarding the methods of interconversion from dynamic FS to shear stress relaxation, both exact conversion methods based on linear viscoelastic theory and approximate conversion methods were used as

mentioned in Table 3. The exact interconversion using the generalized Maxwell model relates the shear relaxation modulus $G(t)$ in time domain to the storage modulus $G'(\omega)$ and loss modulus $G''(\omega)$ in the frequency domain by the following equations.

$$G(t) = \sum_{i=1}^n g_i e^{-t/\lambda_i} \quad (2)$$

$$G'(\omega) = \sum_{i=1}^n g_i \frac{\omega^2 \lambda_i^2}{1 + \omega^2 \lambda_i^2} \quad (3)$$

$$G''(\omega) = \sum_{i=1}^n g_i \frac{\omega \lambda_i}{1 + \omega^2 \lambda_i^2} \quad (4)$$

where g_i and λ_i define the discrete relaxation spectrum and represent the stiffness and relaxation time of the i^{th} Maxwell component. ω is the angular loading frequency from DSR tests. By fitting the dynamic frequency sweep data to Equations 3 and 4, the shear relaxation modulus in time domain can be obtained by Equation 2.

The shear stress relaxation modulus can also be converted from FS data by using the empirical conversion methods. The approximation method proposed by Ninomiya and Ferry (Ninomiya and Ferry 1959) is given in Equation 5.

$$G(t) = G'(\omega) - 0.4G''(0.4\omega) + 0.014G''(10\omega)|_{\omega=2/\pi t} \quad (5)$$

Alternatively, Christensen further simplified the approximate expression as

$$G(t) \approx G'(\omega)|_{\omega=2/\pi t} \quad (6)$$

After obtaining the relaxation modulus curve in the time domain, the m -value is determined as the relaxation rate at the time of interest. Lu *et al.* (Lu et al. 2017) utilized an approximate model originally proposed by Anderson *et al.* (Anderson et al. 1994) to correlate BBR creep stiffness with modulus from DSR. This model requires only the complex modulus and phase angle for the interconversion as shown in Equation 7. The m -value was also empirically approximated as Equation 8 at the temperature and frequency of interest.

$$S(t) = \frac{3G^*(\omega)}{1 + 0.2 \sin(2\delta)} \quad (7)$$

$$m = \delta/90 \quad (8)$$

387 Where $S(t)$ is creep stiffness at time t . δ is phase angle.

388 Similarly, Oshone (Oshone 2018) proposed empirical equations to determine creep stiffness and m -value
389 as shown in Equations 9 and 10.

$$S(t) = 1.28G^*(\omega) + 19.2 \quad (9)$$

$$m = 0.008\delta + 1 \quad (10)$$

390 Equations 7 and 9 are linear functions derived by empirically correlating DSR with BBR testing results.
391 Besides the interconversion process from the frequency domain to time domain, Hajj *et al.* (Hajj et al.
392 2019) also converted the shear response of binders to the uniaxial response using the following equation.

$$S(t) = G(t) (1 + 2\nu) \quad (11)$$

393 where ν is the Poisson's ratio of binder. It was assumed as 0.35 in this study, which was believed to be
394 more reasonable at low temperatures based on the laboratory test results (Benedetto et al. 2007). By doing
395 so, original grading criteria for BBR can be directly applied to the DSR derived parameters. It was
396 reported that the exact interconversion method (Equations 2-4) and approximate conversion method
397 (Equation 5) give an almost identical stress relaxation curve (Sui et al. 2011). For simplicity, the empirical
398 conversion method as shown in Equation 5 was used in this study.

399 *Black diagram*

400 The raw dynamic data of different binders from 4-mm DSR frequency sweep tests were compared in the
401 black diagrams (Figure 7). A black diagram is a useful tool to analyze the rheological data. It can identify
402 possible data discrepancies, verify time-temperature equivalence and thermo-rheological simplicity, and
403 compare different types of bitumen.

404 It can be seen from Figure 7 that there are no obvious discontinuities or sudden changes in the slope of the
405 curves. The smooth curves indicate that 4-mm DSR with the current test configurations can generate

reliable rheological data. The test samples are also confirmed as thermo-rheologically simple. Unlike the black diagrams in the high-temperature range (Wang et al. 2018), the curves of binders in the black space in the low-temperature range do not show any symbolic patterns. Although bitumen with the addition of CRM becomes more elastic as reflected by the shift of rheological data towards lower phase angle (left in the figure), the change in the elastic behavior is not as significant as in the high-temperature range. This is because, at low temperatures, the bitumen matrix is stiffer than rubber particles. From the micromechanical point of view, it has a more dominant impact than rubber particles on determining the rheological properties of CRMB. With the same phase angle value, CRMB binders exhibit lower complex moduli compared to unmodified bitumen. This implies that CRMB may possess improved low-temperature performance than unmodified bitumen, which will be discussed in the following subsections.

Analysis methods for interpreting 4-mm DSR data

There are two main transformations required to obtain BBR parameters from the DSR test results: (a) converting dynamic data from the frequency domain to the time domain, and (b) converting shear response to flexural response. Then, the critical cracking temperatures for low-temperature PG can be determined by the BBR criteria, i.e., $S(t)=300$ MPa and $m=0.30$ at $t=60$ s at PG temperature + 10°C. To demonstrate the calculation process, the neat bitumen 70/100 was taken as an example in this section.

- **Step 1.** The Christensen-Anderson-Marasteanu (CAM) model was used to build the viscoelastic master curves at a reference temperature using the FS data (Figure 8);
- **Step 2.** The shear stress relaxation modulus curve in time domain was converted using the approximation method as given in Equation 5. The master curves of storage modulus and loss modulus were derived by the following relationships.

$$G'(\omega) = |G^*| \sin \delta, \text{ and } G''(\omega) = |G^*| \cos \delta \quad (12)$$

- **Step 3.** Convert the shear relaxation modulus to flexural creep stiffness using Equation 11 as shown in Figure 9.

- **Step 4.** Determine the value of $S(t)$ and m -value at $t= 60$ s through the creep stiffness master curve. The m -value was determined by taking the first derivative after obtaining the trendline equation. The calculated values of S and m are shown in Table 4.
- **Step 5.** Calculate the critical cracking temperatures by interpolation. Steps 1-4 at a different reference temperature were repeated. The selected reference temperatures should bracket specified values of S and m . The critical cracking temperatures were thus determined by interpolating between passing and failing temperatures based on the limiting values of S and m (see Equations 13 and 14).

$$T_{c,S} = T_1 + \frac{(T_1 - T_2)(\log 300 - \log S_1)}{\log S_1 - \log S_2} - 10 \quad (13)$$

$$T_{c,m} = T_1 + \frac{(T_1 - T_2)(0.3 - m_1)}{m_1 - m_2} - 10 \quad (14)$$

where $T_{c,S}$ and $T_{c,m}$ are the critical temperatures or true grade controlled by the creep stiffness and m -value respectively. T_1 is the temperature at which S or m passes the criterion while T_2 is the temperature at which S or m fails the criterion. S_1 and S_2 are the creep stiffness (MPa) at T_1 and T_2 . m_1 and m_2 are the creep rate at T_1 and T_2 .

Critical cracking temperature and Delta T_c parameter

Using the above calculation process, the derived values of S and m from 4-mm DSR test results at different temperatures are summarized in Table 4. In general, binders with a lower creep stiffness and a higher creep rate have improved resistance to thermal cracking. The values of S and m highlighted in bold and italic in Table 4 are the data that bracket the specifications and are used to calculate the critical cracking temperatures (Equations 13 and 14). It can be seen from Table 4 that there are no consistent trends for the change in creep stiffness and creep rate of different binders at different temperatures. At the temperatures that bracket the specifications (shaded data), the addition of wax-based additives to neat bitumen increased the creep stiffness and decreased the creep rate. This is because the wax may crystallize in binders at low temperatures, resulting in a stiffening effect. By contrast, the chemical-based additive

decreased the stiffness and increased the creep rate of the neat bitumen. The polymers and oils in the chemical additive may be conducive to improve the cracking resistance. Similar effects of warm-mix additive were found on CRMB. Briefly, wax-based additives negatively affect the thermal cracking performance of binders while chemical-based additives can improve the low-temperature performance. Comparing the neat bitumen with CRMB binders, rubber modification decreases the stiffness and increases the creep rate at low temperatures. Further increasing the rubber content, the improvement of low-temperature performance is more prominent. The above findings are consistent with previous BBR test results for characterizing the low-temperature performance of CRMB binders (Akisetty et al. 2010).

The low PGs of different binders based on the current PG specifications are also shown in Table 4. The addition of warm-mix additives does not change the low-temperature PG of neat bitumen as -28°C. The modification by CRM further decreases the low-temperature PG. When increasing the CRM content to 22%, the low PG of CRMB is improved to -40°C compared to other CRMBs with lower CRM contents which have a low PG of -34°C. The wax-based additive increases the continuous low PG of CRMB by 1.1°C while CRMB-22 with the chemical-based additive decreases the continuous low PG by 1.8°C.

Since the current PG grading system adopts an increment of 6°C, binders having the same PG may have a slightly different performance at a certain temperature. Therefore, the critical temperatures (true continuous grade) of different binders for low-temperature PG calculated using Equations 13 and 14 are summarized in Table 5. To account for the different governing mechanisms for the performance grade of a binder, a new parameter called ΔT_c was defined below

$$\Delta T_c = T_{c,s} - T_{c,m} \quad (15)$$

The sign of ΔT_c indicates whether the performance grade of the binder is governed by its creep stiffness ($+\Delta T_c$) or creep rate ($-\Delta T_c$). The absolute magnitude of ΔT_c indicates the degree to which the binder is governed by either creep stiffness or creep rate. ΔT_c values for different binders were shown in Table 5.

It can be seen from Table 5 that both $T_{c,S}$ and $T_{c,m}$ of CRMB gradually decrease as the increase of rubber content, indicating a lower low-temperature PG. With regard to the effect of warm-mix additives, the addition of wax-based additive increases the critical temperatures of both neat and rubber modified binders while chemical-based additive slightly decreases the critical temperature. In addition, it is noteworthy that the $T_{c,S}$ of 70/100 based binder (with/without warm additives) is higher than the corresponding $T_{c,m}$, resulting in a positive value of ΔT_c . By contrast, CRMB based binder has the opposite situation in which its $T_{c,S}$ is lower than $T_{c,m}$. However, there is not a clear trend of the effects of rubber content and warm additives on the value of ΔT_c . The different signs of ΔT_c of neat bitumen and CRMB implies that the low-temperature PG of 70/100 based binders are creep stiffness controlled while CRMB binders are m -value controlled. Creep stiffness does not present a complete picture of binder cracking tendency at low temperatures. This is because bitumen is a viscoelastic material, which is able to relax applied stresses. In other words, if given sufficient time, bitumen will shed the built-up stresses when applying a load or changing the temperature condition. This ability to relax the stresses of binders is defined as creep rate or m -value in the BBR test.

Understanding the different controlling mechanisms can have two benefits in optimizing the binder design. Firstly, neat bitumen is more suitable in the climates where rapid changes to cold temperatures are often observed. This is because high m -value CRMB binders may encounter a rapid increase in thermal stress that can lead to cracking before relaxation can occur (Marasteanu and Cannone Falchetto 2018). CRMB is more suitable for climates where cold temperatures stay for an extended period because more relaxation of CRMB takes place due to its higher m -value. Secondly, to further improve the low-temperature performance grades of binders, different strategies should be used for neat bitumen and CRMB. For neat bitumen, emphasis should be put on decreasing the creep stiffness while for CRMB, efforts should be done in increasing the creep rate.

Modification Mechanism for the High- and Low-temperature Performance

CRMB can be regarded as a binary composite in which rubber particles are embedded in the bitumen matrix. To figure out how the rubber modification and warm-mix additives influence the binder performance, the bitumen and rubber phases of CRMB-22, CRMB-22-W and CRMB-22-C were separated, and their rheological properties were investigated through frequency sweep tests. In general, bitumen-rubber interaction controls the binder properties through the following ways: (1) changing the mechanical properties of bitumen matrix due to the loss of light fractions absorbed by rubber and the potential released components from rubber; (2) changing the mechanical properties of rubber due to rubber swelling; (3) changing the volume fraction of rubber due to swelling (Wang et al. 2020). With the obtained viscoelastic data at various frequencies and temperatures, master curves were established based on the time-temperature superposition principle (TTSP).

Effect of Rubber Modification

Figure 10 presents the viscoelastic master curves of bitumen and rubber phases at a reference temperature of 30°C. Firstly, comparing neat bitumen 70/100 and CRMB-22, CRMB-22 has higher complex moduli than 70/100 at low frequencies (high temperatures) and lower complex modulus at high frequencies (low temperatures). In the whole frequency range, the phase angle of CRMB-22 is lower than that of 70/100, indicating more elastic behaviors. Considering low/high frequencies correspond to high/low temperatures in the frame of master curves, CRMB-22 is stiffer than 70/100 at high temperatures while softer at low temperatures. This coincides with the finding that CRMB-22 has improved both high-temperature rutting and low-temperature cracking performance. Secondly, it can be found that the bitumen phase CRMB-22-BP is stiffer and more elastic than neat bitumen 70/100 because of the loss of light components absorbed by rubber particles, which increases the proportions of asphaltenes in bitumen. Asphaltenes are primarily responsible for the increase of complex modulus and decrease of phase angles (Apostolidis et al. 2017). Thirdly, comparing the bitumen phase (CRMB-22-BP) with the swollen rubber phase, the swollen rubber sample has lower moduli at high frequencies and higher moduli at low frequencies. This means CRMB-22 will have a smaller creep stiffness and a higher creep rate than the neat bitumen at low temperatures,

which is exactly the same as measured in the 4-mm DSR tests. Standing from a micromechanics point of view, the property changes of the bitumen phase and the inclusion of swollen rubber particles in the bitumen matrix together contribute to the peculiar viscoelastic response of CRMB-22, i.e., stiffer and more elastic at high temperatures, softer and more elastic at low temperatures. This finding was also verified by the authors using micromechanical modelling (Wang et al. 2020). These peculiar viscoelastic responses of CRMB-22 explain why CRMB in general has improved resistance to both rutting and thermal cracking.

Effect of Warm-mix Additives

The warm additives were added to CRMB binders at a relatively low temperature after the bitumen-rubber mixing process. Therefore, the additives mainly influence the properties of the bitumen matrix instead of the rubber phase (Yu et al. 2017). Figure 11 presents the viscoelastic master curves of bitumen phases containing warm-mix additives at a 30°C. For neat bitumen 70/100, the addition of wax-based additives resulted in a significant increase in complex modulus and reduction in phase angle. The reason has been explained before, which is due to the stiffening effect of wax. By contrast, the addition of chemical-based additives seemed to slightly soften the binder. Chemical-based additives had insignificant effects on the viscoelastic properties of binders. Comparing the bitumen phases of CRMB binders with and without additives, similar effects of warm additives on the properties of bitumen phases were observed. The findings from the viscoelastic master curves further verify the conclusions in the previous sections and reveal how additives influence the high- and low-temperature performance of binders. Based on the above analysis, it should be noted that although warm-mix additives can be used to effectively reduce the viscosity of CRMB and hence reduce the construction temperatures, the type of additives should be cautiously selected to ensure the high- and low-temperature performance are not compromised.

CONCLUSIONS AND RECOMMENDATIONS

The present study investigated the high- and low-temperature performance of warm crumb rubber modified bitumen (CRMB) binders using MSCR and low-temperature FS tests. The obtained relaxation parameters from 4-mm DSR tests were used to determine the low-temperature PG by directly applying the

BBR criterion. Two transformations were done in this process, namely converting viscoelastic master curves from the frequency domain to the shear relaxation modulus in the time domain, and converting shear response to flexural response by assuming a constant Poisson's ratio. The rheology of the bitumen and rubber phases of CRMB was investigated to gain insights into the rubber and additives modification mechanism and its impact on the binder performance. The following conclusions can be drawn:

- CRMB binders have superior rutting and thermal cracking resistance due to rubber modification. The improvement of high- or low-temperature performance is more prominent at higher rubber concentrations.
- Warm-mix additives have different effects on high- or low-temperature performance. Generally, the wax-based additive improves the rutting resistance while having adverse effects on the low-temperature performance. By contrast, chemical-based additive slightly impairs the high-temperature rutting resistance while improving the low-temperature performance.
- In terms of the critical cracking temperature, the low-temperature PG of 70/100 based binders is creep stiffness controlled while CRMB binders are *m*-value controlled. Rubber modification changes the controlling mechanism of low-temperature performance.
- The stiffening of the bitumen phase and the inclusion of swollen rubber particles in the bitumen matrix together contribute to the peculiar viscoelastic response of CRMB, i.e., stiffer and more elastic at high temperatures, softer and more elastic at low temperatures. This modification mechanism explains the superior rutting and thermal cracking resistance of CRMB.
- Although warm-mix additives can effectively reduce the viscosity of CRMB and hence reduce the construction temperatures, the type of additives should be cautiously chosen to ensure high- and low-temperature performance are not compromised.

In future studies, BBR tests on binders can be performed to further verify the conclusions from 4-mm DSR tests. High- and low-temperature performance of asphalt mastic and mixture are recommended to be investigated to verify the findings at the binder level.

DATA AVAILABILITY STATEMENT

All data, models, and code generated or used during the study appear in the submitted article.

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Table 1. One-way ANOVA results between different replicates of CRMB-22.

Source of variation		<i>SS</i>	<i>DF</i>	<i>MS</i>	<i>F</i>	<i>P-value</i>	<i>F-critical</i>
MSCR	Shear strain	40993.33	2	20496.67	0.3387	0.7127	2.9972
4-mm DSR FS	Complex modulus	6.82E+09	2	3.41E+09	0.2667	0.7669	3.1588
	Phase angle	0.0197	2	0.0098	0.0021	0.9979	3.1682

*Note: SS=Sum of Squares, DF=degree of freedom, MS= Mean Square.

Table 2. Summary of MSCR results of different binders at the short-term aged state.

Binder type	<i>J_{nr}</i> (1/kPa)		<i>J_{nr}diff</i> (%)	<i>J_{nr}slope</i> (%)	<i>R</i> (%)		Traffic level*
	0.1 kPa	3.2 kPa			0.1 kPa	3.2 kPa	
70/100	3.489	3.938	12.87	14.48	1.55	0.00	S
70/100-W	0.602	2.478	311.63	60.52	45.78	0.18	S
70/100-C	3.323	3.778	13.69	14.68	1.52	0.00	S
CRMB-5	1.336	1.780	33.23	14.33	22.35	3.73	H
CRMB-10	0.466	0.914	96.14	14.45	48.12	15.47	V
CRMB-15	0.142	0.413	192.86	8.71	76.32	32.45	E
CRMB-22	0.022	0.182	727.27	5.16	91.44	41.66	E
CRMB-22-W	0.019	0.176	826.32	5.06	93.42	45.69	E
CRMB-22-C	0.046	0.292	534.78	7.94	86.57	35.26	E

*Note: S=Standard, H=Heavy, V=Very Heavy, E=Extremely Heavy.

Table 3. Methods and criteria for low-temperature grading binders using 4-mm DSR.

Reference	Conversion methods	Creep stiffness grading criterion	<i>m</i> -value grading criterion
Sui et al. 2011	Generalized Maxwell model or Ninomiya and Ferry approximation method	$G(t) < 180 \text{ MPa}$ at $t=7200 \text{ s}$ at actual PG temperature	$m > 0.26$ from $G(t)$ at $t=7200 \text{ s}$ at actual PG temperature
Farrar et al. 2015	Christensen approximation method	$G(t) < 143 \text{ MPa}$ at $t=60 \text{ s}$ at PG temperature + 10°C	$m > 0.28$ from $G(t)$ at $t=60 \text{ s}$ at PG temperature + 10°C
Lu et al. 2017	Empirical Equation 7	$S(t) < 143 \text{ MPa}$ at $t=60 \text{ s}$ at PG temperature + 10°C	$m > 0.28$ at $\omega = 2/\pi t$ at PG temperature + 10°C
Oshone 2018	Empirical Equation 9	NA	NA
Hajj et al. 2019	Ninomiya and Ferry approximation method	$S(t) < 300 \text{ MPa}$ at $t=60 \text{ s}$ at PG temperature + 10°C	$m > 0.30$ from $S(t)$ at $t=60 \text{ s}$ at PG temperature + 10°C

Table 4. Creep stiffness and m -value derived from 4-mm DSR test results.

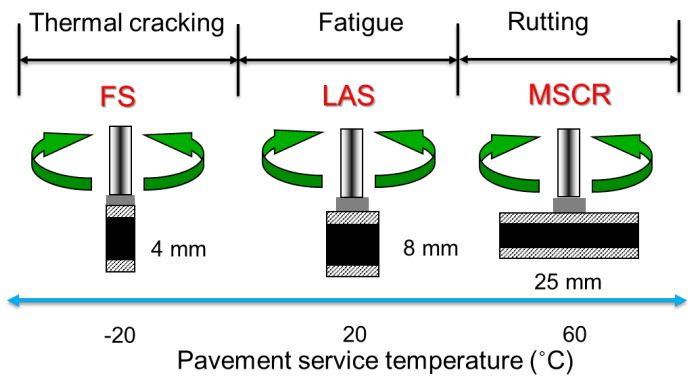
Binder type	-18°C		-24°C		-30°C		-36°C		Continuous low PG (°C)	Low PG (°C)
	S (MPa)	m	S (MPa)	m	S (MPa)	m	S (MPa)	m		
70/100	174.7	0.35	402.4	0.28	785.2	0.20	1112.4	0.16	-31.9	-28
70/100-W	216.1	0.33	625.9	0.24	1283.8	0.15	1453.9	0.13	-29.9	-28
70/100-C	161.7	0.37	466.0	0.28	1092.8	0.17	1394.4	0.13	-31.5	-28
CRMB-5	107.1	0.38	253.2	0.31	524.7	0.24	797.2	0.19	-34.7	-34
CRMB-10	92.0	0.39	223.9	0.32	469.6	0.25	723.8	0.20	-35.7	-34
CRMB-15	70.9	0.40	163.2	0.33	333.6	0.27	595.7	0.21	-37.1	-34
CRMB-22	56.0	0.42	124.9	0.36	252.3	0.30	453.9	0.24	-40.4	-40
CRMB-22-W	66.1	0.42	156.9	0.35	277.4	0.29	459.2	0.23	-39.3	-34
CRMB-22-C	61.2	0.43	134.1	0.37	237.7	0.31	443.9	0.26	-42.2	-40

Table 5. Critical cracking temperatures and ΔT_c values for different binders.

Binder type	70/100	70/100-W	70/100-C	CRMB-5	CRMB-10	CRMB-15	CRMB-22	CRMB-22-W	CRMB-22-C
$T_{c,s}$	-31.9	-29.8	-31.5	-35.4	-36.4	-39.1	-41.8	-40.9	-42.2
$T_{c,m}$	-32.2	-30.1	-32.5	-34.7	-35.7	-37.1	-40.4	-39.3	-41.1
ΔT_c	0.3	0.3	1.0	-0.7	-0.7	-2.0	-1.4	-1.6	-1.1

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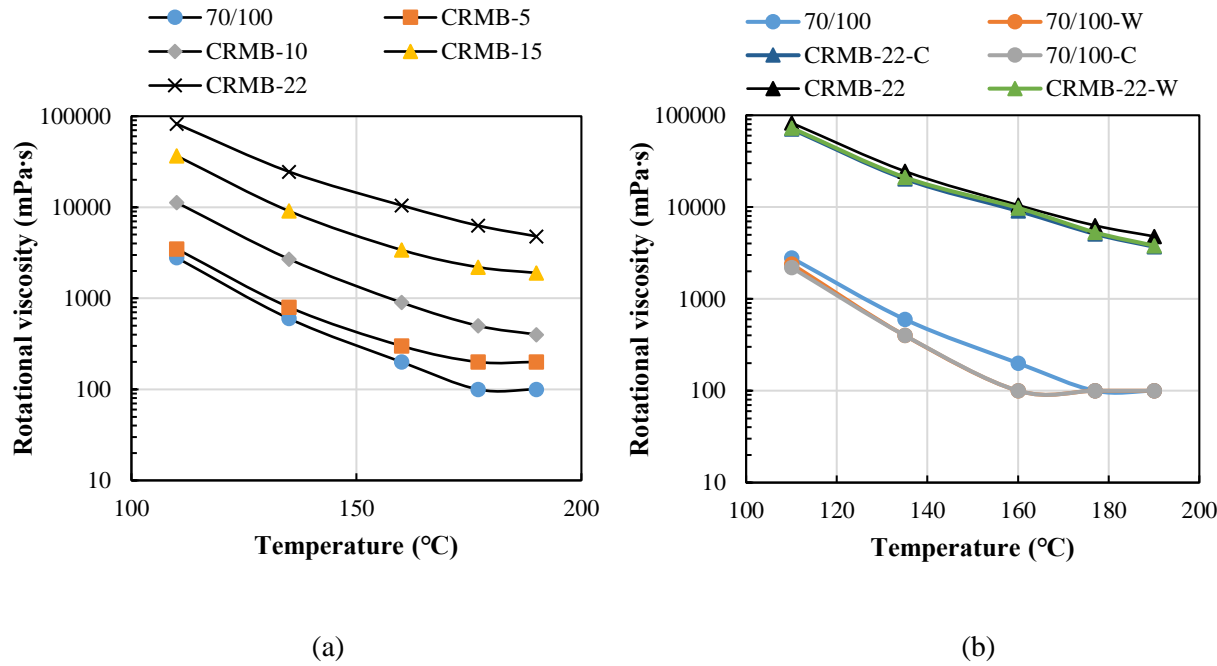
4

Figure 1. Unified DSR method for binder performance characterization from low to high temperatures.

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Figure 2. Rotational viscosities of CRMB binders with (a) different rubber contents; (b) warm-mix additives.

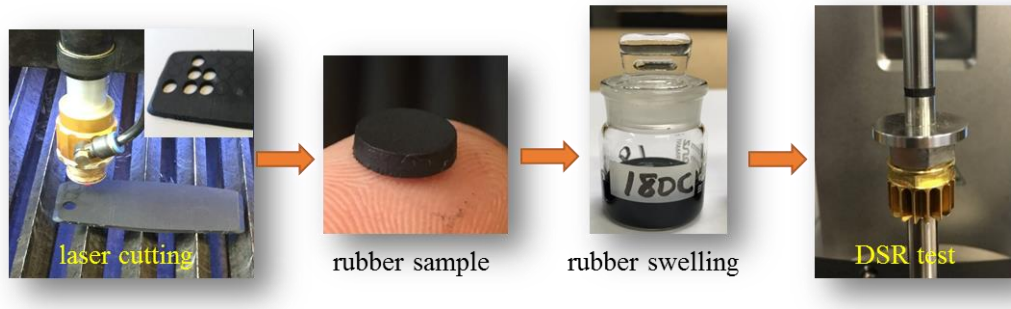


Figure 3. Dry and swollen rubber samples for DSR tests.

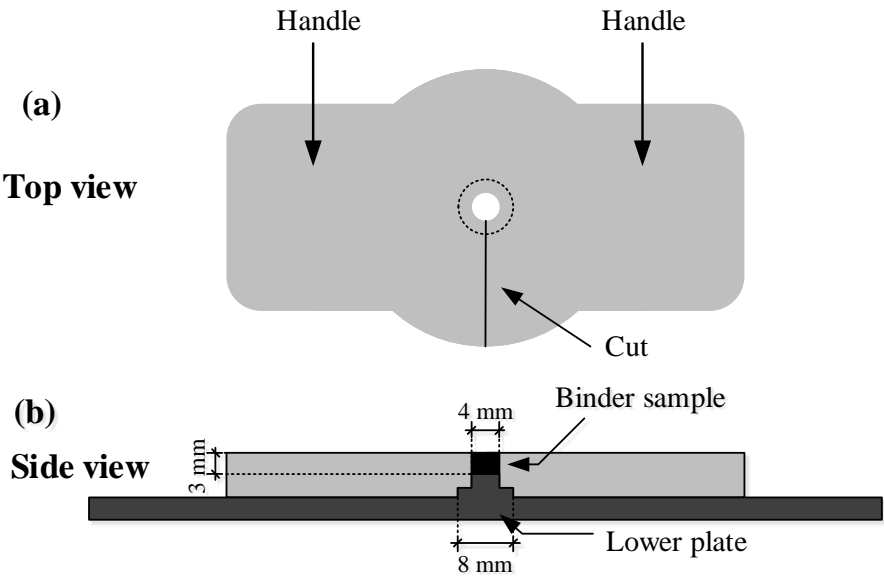
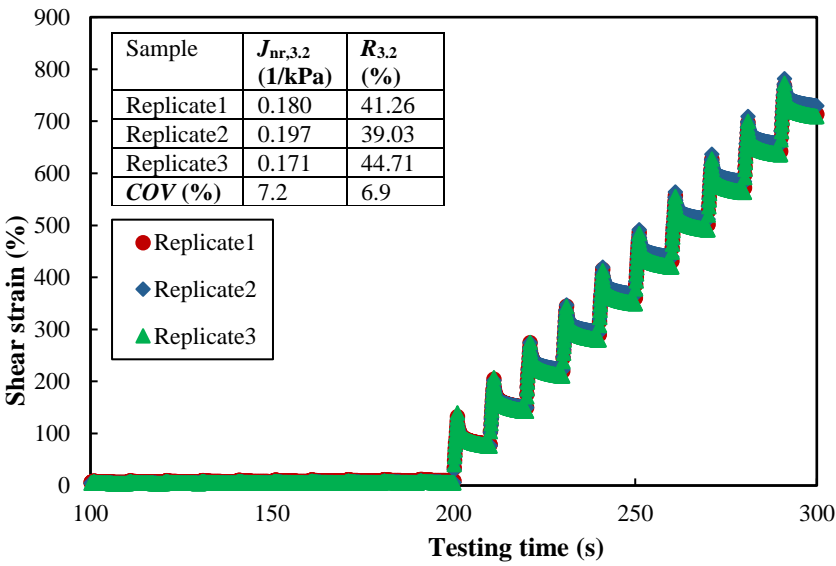
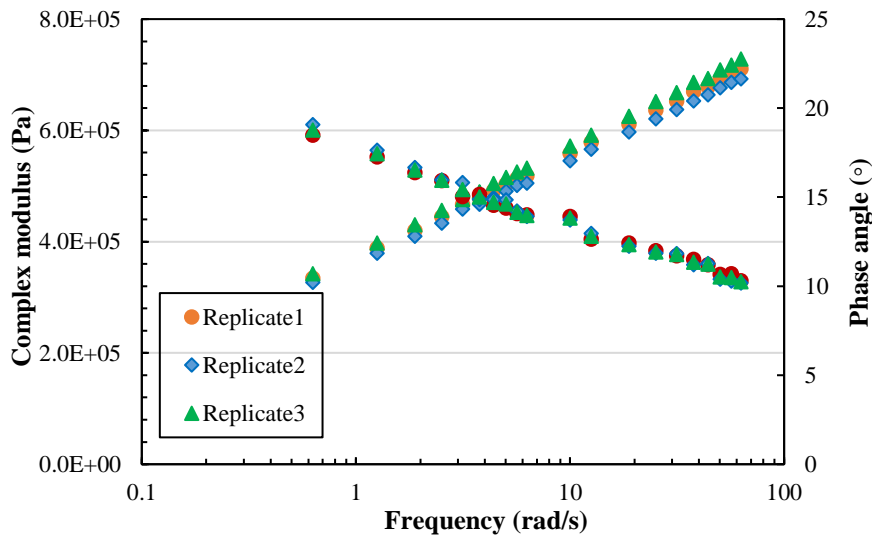


Figure 4. The butterfly silicone mold for 4-mm DSR sample installation: (a) top view; (b) side view.

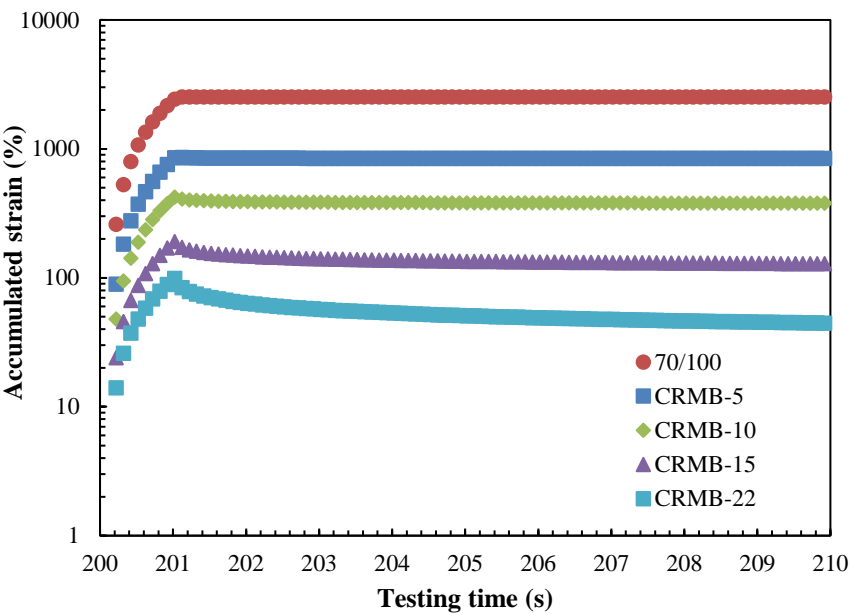


(a)

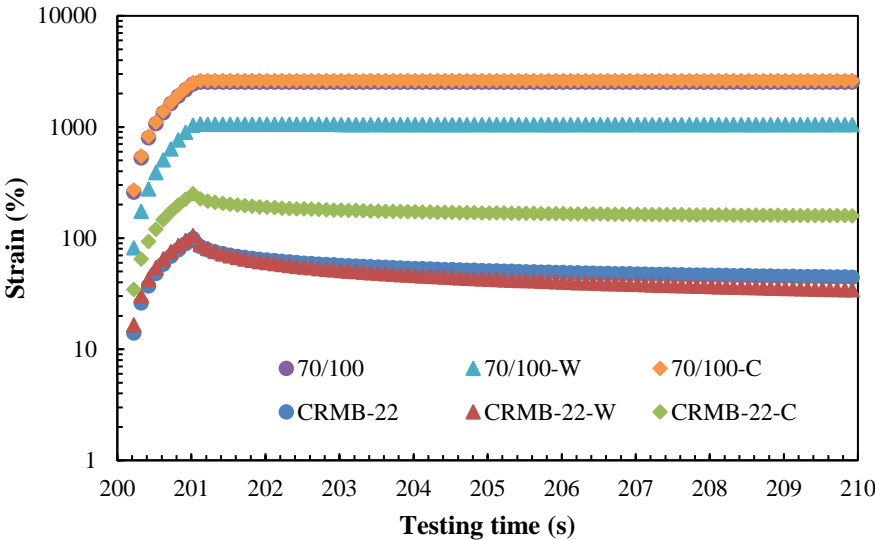


(b)

Figure 5. Variability of binder test results from CRMB-22 samples: (a) MSCR test using 25-mm plates at 64 °C; (b) Frequency sweep test using 4-mm plates at -30 °C. (COV=coefficient of variation)

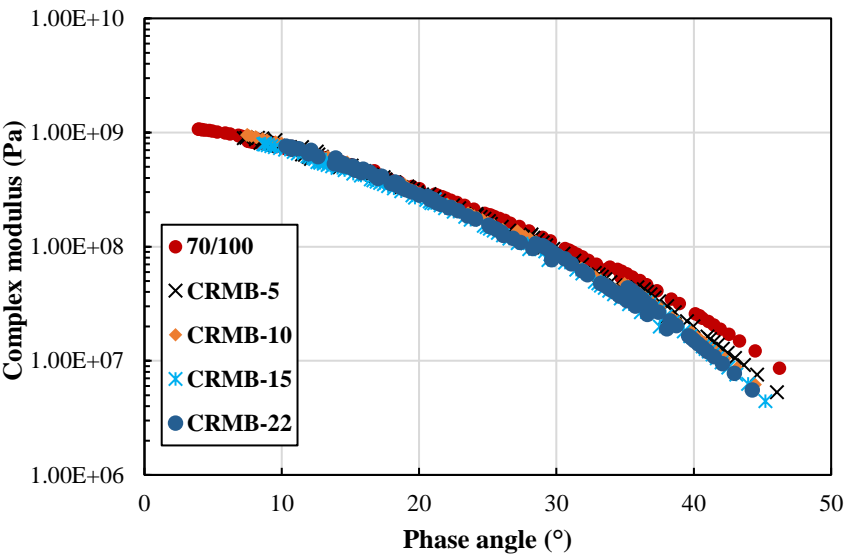


(a)

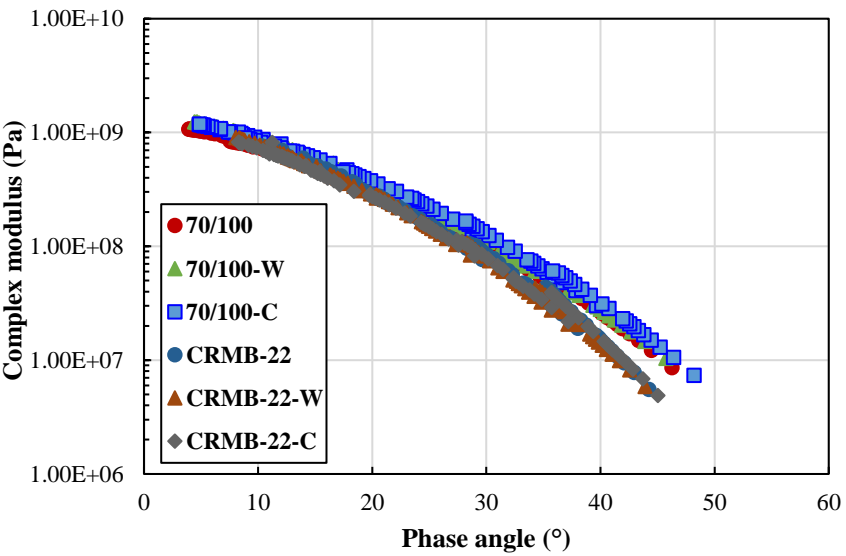


(b)

Figure 6. Creep and recovery strain responses of different binders at 3.2 kPa: (a) CRMB with different rubber contents; (b) Binders with warm-mix additives.

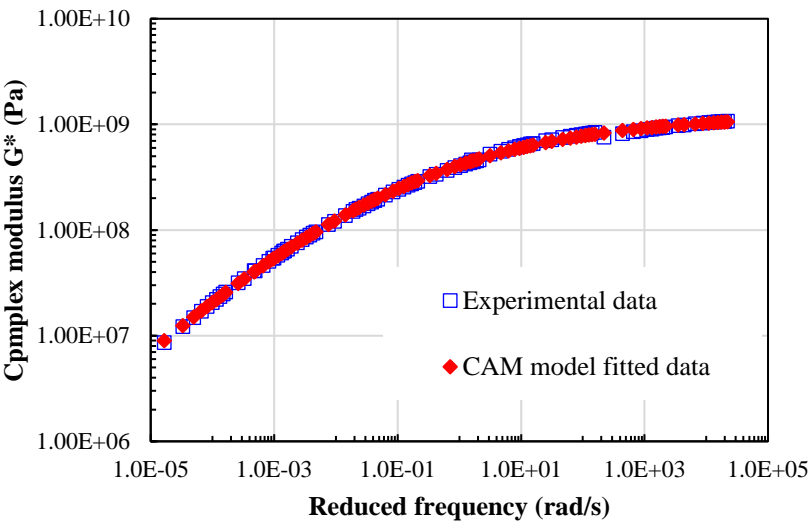


(a)

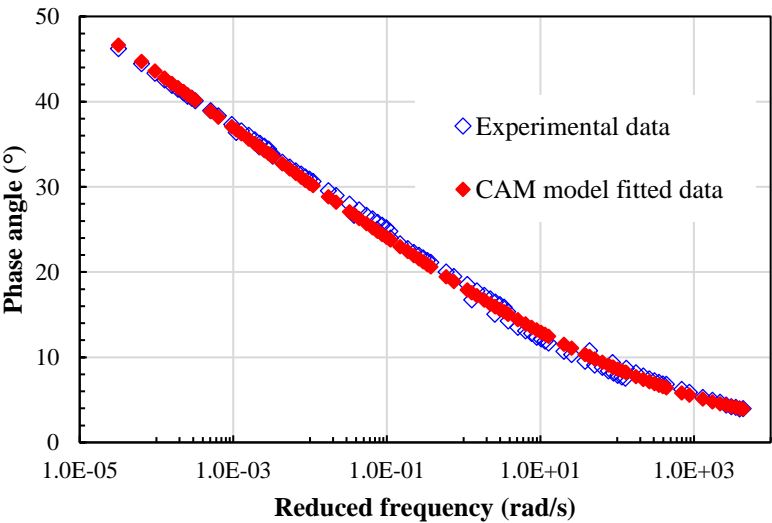


(b)

Figure 7. Black diagram of (a) CRMB binders and (b) binders with warm-mix additives.



(a)



(b)

Figure 8. (a) Complex modulus and (b) phase angle master curves of neat bitumen 70/100 at a reference temperature of -18 °C.

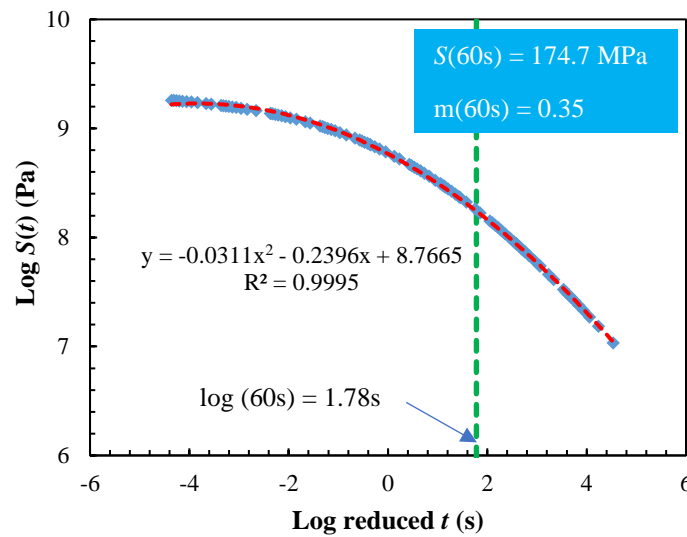
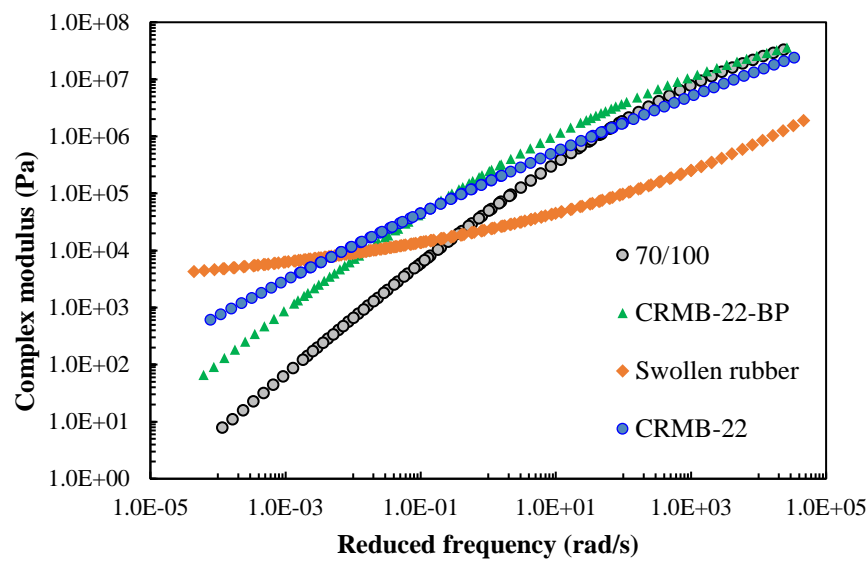
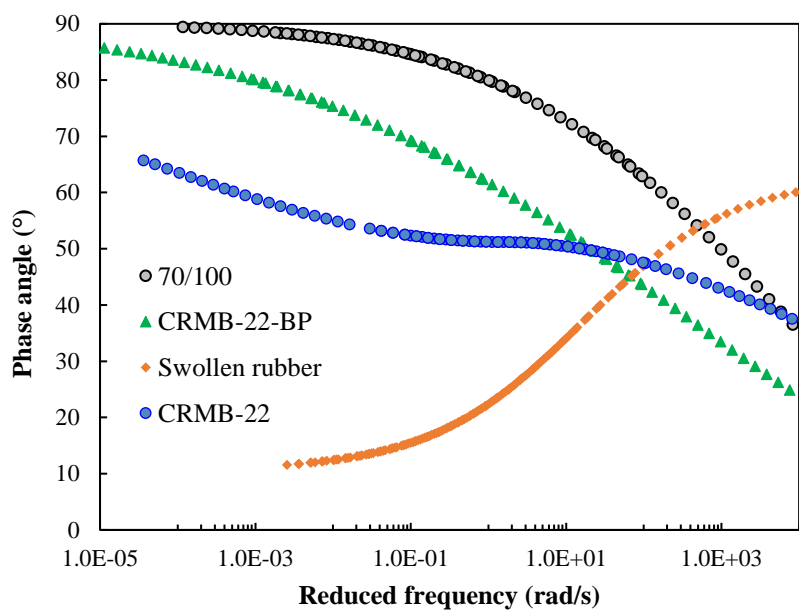


Figure 9. Master curve of creep stiffness at a reference temperature of -18 °C.

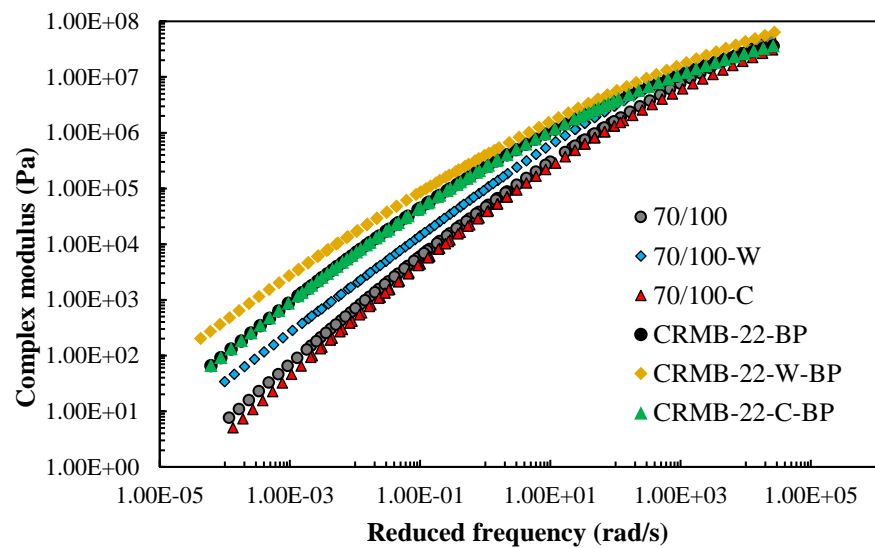


1 (a)

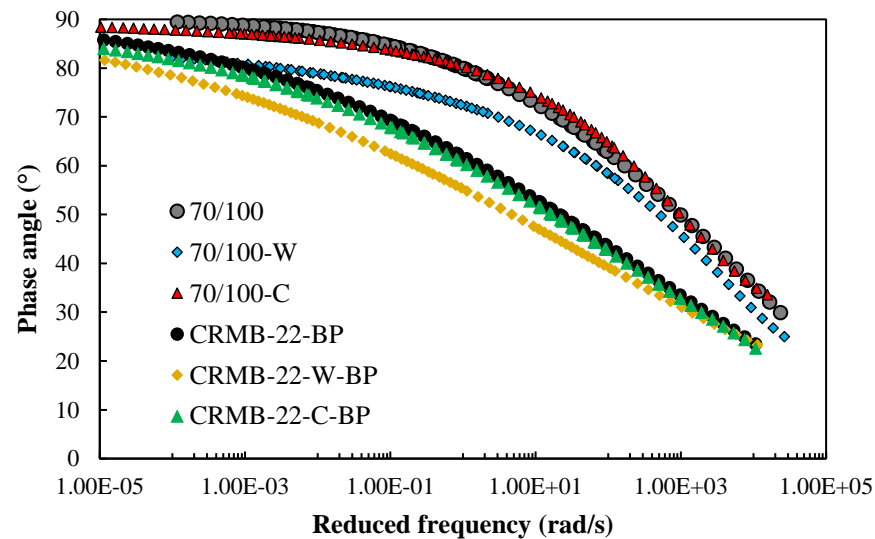


2 (b)

3 **Figure 10.** Viscoelastic master curves of bitumen and rubber phases: (a) complex modulus and (b) phase
4 angle.



1 (a)



2 (b)

3 **Figure 11.** Viscoelastic master curves of bitumen phases containing warm-mix additives: (a) complex
4 modulus and (b) phase angle.