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# Black Space Rheological Assessment of Asphalt Material Behavior

#### Reference

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#### ABSTRACT

 $C_{74}$ Black Space diagrams representing rheological data of asphalt materials in the form of complex modulus ( $|G^*|$  or  $|E^*|$ ) versus phase angle ( $\delta$ ) have been successfully used for interpretation of material behavior and performance. Previous studies have used Black Space for 12 identification of testing geometry compliance errors when testing over multiple temperatures 13 and loading times (frequencies), screening of the "thermo-rheological simplicity" of various 14 binders and mixtures, and detailed evaluation of the performance balance in term of "stiffness" 15 versus "relaxation" needs. This paper provides an overview of how Black Space can be further 16 used to provide a greater understanding of the concepts of damage and healing and cracking 17 susceptibility and fracture, and to also quantify the complex rheological response of alternative 18 binders. In terms of the damage assessment, cyclic loading tests were analyzed using Black 19 Space to identify additional physical phenomena such as nonlinearity, self-heating, and thix-20 otropy. The cracking analysis has included thermal, fatigue, and durability cracking as well as 21 the use of Black Space to access the performance of asphalt mixtures subjected to aging as 22 well as rejuvenation and materials with recycled asphalt. Concepts such as the Glover-Rowe 23 parameter that are based around Black Space and linked to other forms of rheological indices 24 such as the low-temperature stiffness and relaxation rate parameters are introduced. The re-25 sults in the paper show that Black Space provides a critical means of rheological characteri-26 zation to investigate and evaluate the properties and performance of both binders and 27 mixtures. This is particularly relevant at a time when there is a concerted move within the 28 asphalt paving industry toward more sustainable solutions and increased demand for reuse 29 and recycling of materials in asphalt mixtures. 30

#### Keywords

black space, rheology, cracking, damage, aging, rejuvenation, alternative binders

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## Introduction

The linear viscoelastic (LVE) characterization of asphalt materials has been extensively used in material selection, AQ6 mixture design, pavement design, and performance prediction. As a viscoelastic material, asphalt binder exhibits 35 both elastic and viscous components of response and displays a temperature-, time-, and history-dependent re-36 lationship between applied stresses and resultant strains.<sup>1,2</sup> However, within the LVE region of response, the 37 interrelation between stress and strain is influenced by temperature, loading history, and time alone, not by 38 the magnitude of the stress (i.e., deformation at any time and temperature is directly proportional to the applied 39 load).<sup>3</sup> In addition, as asphalt binder is responsible for the viscoelastic behavior of all asphalt materials, it plays a 40 dominant role in defining many of the aspects of asphalt road performance such as strength and stiffness, per-41 manent deformation, and cracking. 42

Various techniques and methodologies exist to measure the rheological (viscoelastic) properties of asphalt 43 binders including dynamic mechanical analysis using oscillatory testing generally by means of a dynamic shear  $\bigcirc$ rheometer (DSR).<sup>4-6</sup> The principal viscoelastic parameters that are obtained from the DSR are the complex shear 45 modulus (G\*) and the phase angle ( $\delta$ ). Absolute value of G\* (|G\*|) is defined as the ratio of maximum (shear) 46 stress to maximum strain and provides a measure of the total resistance to deformation (stiffness) when the 47 bitumen is subjected to shear loading. G\* contains elastic and viscous components that are defined as the storage 48 modulus (G') and loss modulus (G'), respectively. These two components are related to the complex modulus 49 and to each other through the phase (or loss) angle ( $\delta$ ), which is the phase, or time, lag between the applied shear 50 stress and shear strain responses during a test. The phase angle is a measure of the viscoelastic balance of the 51 material behavior, with extreme values of 90° corresponding to purely viscous response and 0° to purely elastic 52 behavior, and provides an indication of the potential stress relaxation of the material. Between these two extremes, 53 the material behavior can be considered viscoelastic in nature with a combination of viscous and elastic responses. 54 Unlike oscillatory shear testing of binders, asphalt mixture complex modulus is typically measured in extensional 55 mode through uniaxial loading on a cylindrical specimen; the outcome of this test is either one- or three-dimen-56 sional LVE characterization in the form of either complex modulus ( $E^*$ ) or both  $E^*$  and  $\nu^*$  (where  $\nu^*$  is complex 57 Poisson's ratio). 58

One of the primary techniques used in analyzing dynamic oscillatory data involves the construction of 59 master curves using the interrelationship between temperature and frequency (time) to produce a continuous 60 rheological parameter curve at a reduced frequency or time scale. The principle used to relate the equivalency 61 between time and temperature and thereby produce a master curve is known as the time-temperature super-62 position principle or the method of reduced variables.<sup>1</sup> The production of a smooth, continuous master curve 63 generally relies on the asphalt binder exhibiting simple rheological behavior, termed or classified as "thermo-64 rheological simplicity." An alternative to the production of master curves is to present the rheological data 65 in the form of a Black Space diagram. Black Space provides a two-dimensional representation of the two fun-66 damental characteristics of LVE response, namely the norm of complex modulus ( $|G^*|$  or  $|E^*|$ ) and the phase 67 angle ( $\delta$ ), at each of the temperatures and loading frequencies at which these parameters are measured. This two-68 dimensional representation allows for easy visualization of a material's response in the context of its stiffness as 69 well as relaxation capabilities and more importantly, the interplay between them at any given loading frequency or 70 temperature. 71

Airey<sup>7</sup> demonstrated how Black Space could be used to not only represent the viscoelastic properties of 72 different asphalt materials as an alternative to master curves and isochronal and isothermal plots, but also 73 how to use Black Space to identify testing issues with the rheological data, performance issues with the rheological 74 data, or both. Black Space was used to help identify compliance (testing) errors in rheological data associated with 75 the inappropriate use of various DSR spindle geometries. Black Space diagrams were also shown to be an 76 extremely powerful means of tracking the rheological changes in asphalt binders associated with both short-77 and long-term aging as well as means of studying the rheological properties of complex binders such as modified 78 binders. Since then, the use of Black Space has been explored by a number of researchers. 79

This paper describes selected recent developments in the use of Black Space diagrams as a means of assessing the performance of distress mechanisms associated with asphalt materials in the following ways: the use of Black Space in aiding the interpretation and understanding of key areas consisting of damage and healing assessment of asphalt materials; the use of rheological parameters to assess cracking susceptibility; and finally, the rheological characterization of alternative binders are described with reference to prominent studies in each of these areas. It is important to note that the paper is not intended to be a comprehensive review of all the possible uses of Black Space in asphalt technology literature and is simply an overview of some key examples, specifically those focusing on newer techniques to leverage Black Space for improved asphalt material performance and increased level of sustainability. 87

## Rheological Assessment of Global Damage and Healing

Black Space diagrams have been successfully used to provide a framework for the interpretation of experimental89results from cyclic loading and fatigue tests.8.9Using Black Space, changes that induce complex modulus var-90iations during loading can be investigated based on different physical phenomena including nonlinearity, self-91heating, thixotropy, and damage. Some of these can be considered reversible and therefore produce biasing effects92with respect to damage analysis.93

#### NONLINEARITY EFFECTS

During fatigue tests, nonlinearity can be observed on the complex modulus at the beginning of cyclic loading95where at a fixed temperature and frequency, the measured complex modulus depends on the applied axial strain96amplitude. This alteration in complex modulus is instantaneous and reversible. This phenomenon can be studied97independently from damage effects provided that the number of cycles remains low.98

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An example of nonlinearity effects on complex modulus is given in figure 1 where the nonlinearity effect on 99 asphalt mixtures was studied by means of cyclic loading tests where strain amplitude was increased and then 100 decreased, linearly, from 10  $\mu$ m/m to 110  $\mu$ m/m. During the test, temperatures ranged from 8°C to 14°C and 101 frequencies from 0.3 to 10 Hz.<sup>10</sup> Inside this narrow range, the nonlinearity direction determined on the Black 102 Space diagram was not found to be constant, although variations were small. 103

#### THIXOTROPY AND LOCALIZED HEATING EFFECTS

Self-heating (as well as self-cooling during rest periods) will also affect the complex modulus during cyclic loading105because of viscous behavior causing heat dissipation. Asphalt materials are thermosensitive; therefore, changes in106complex modulus occur during loading because of self-heating temperature increases. During rest periods, the107specimen temperature will tend to initially increase and then subsequently decrease. The change in complex108modulus because of temperature variation is once again reversible.109

Thixotropy effects on the complex modulus are observed during loading and resting and defined as 110 a progressive decrease in modulus with time under loading and a gradual recovery when loading is removed 111 at a constant temperature. A classical explanation for this phenomenon is the buildup or the destruction of 112 a structural network in the material under loading and resting. During loading, stiffness tends to decrease, while during rest periods it increases up to an equilibrium value with the phenomenon being reversible. Finally, damage 114 affects the complex modulus during loading with material cross-section reducing (microcracks) and a subsequent 115 reduction in stiffness. With damage accumulation, microcracks may coalesce into a macrocrack resulting in the eventual material failure with this phenomenon being irreversible. 117

The evolution of complex modulus during cyclic loading and rest periods can be investigated in complex 118 representations such as Black Space diagrams.<sup>9</sup> Figure 2 represents an example of test results and an interpretation of the evolution of the effects of each of the phenomena described above. In Black Space, it is seen that each 120 phenomenon presents a particular direction of complex modulus evolution. Before performing the continuous 121 cyclic loading test, information on temperature-sensitivity may be obtained from classical complex modulus tests. 122 If these tests include different strain amplitudes, nonlinearity effects can also be calculated. 123

#### FIG. 1

(A) Scheme of nonlinearity direction in Black Space and definition of  $p_E$  and  $p\phi$ coefficients used to quantify the effects of nonlinearity on complex modulus (norm and phase angle respectively) and (B) results obtained from nonlinearity tests (linear increase and decrease of the amplitude of strainloading cycles from 10 to 110  $\mu$ m/m) at different temperatures and frequencies.10



To observe damage, tests with loading and rest periods should be undertaken. After each rest period, the 124 complex modulus can be compared with its initial value. Obviously, the comparison should consider the potential 125 nonlinearity effects (strain amplitude) as well as temperature effects. Some cracks developed during loading could 126 also heal during rest periods and make the damage evaluation false. However, the healing phenomenon is expected to occur more slowly than other reversible effects (temperature and thixotropy). 128

Babadopulos et al.<sup>11</sup> suggested that damage should have no effect (or negligible effect) on phase angle. Phase 129 angle recovers quickly during rest periods, which indicates that its change is mainly because of reversible effects 130 (temperature and thixotropy). Loading and rest periods tests were performed on bitumen to differentiate each of 131 these effects as shown in figure 3, where after 10,000 cycles of loading at a 2 % shear-strain amplitude, specimens 132

#### FIG. 2

Directions of different phenomena on Black Space<sup>9</sup>: example of experimental results.



were provided with 4-h rest periods. During the rest periods, complex modulus was measured intermittently with 133 0.05 % strain amplitude cycles. To separate the different effects, data were corrected from temperature evolution 134 effects and plotted in figure 3 at a fixed temperature ( $T_{ini}$ ) of 11°C. 135

The nonlinearity effect is visible between the complex modulus measured at low strain at the end of the rest 136 periods and the complex modulus measured (after three cycles) at high amplitude at the beginning of the next 137 loading period. The damage created by the loading periods can be seen as the difference between low-strain 138 complex modulus measured at the end of the rest periods. Considering experimental fluctuations, phase angle 139

#### FIG. 3

Loading and rest periods test results corrected from temperature effects at  $T_{ini} = 11^{\circ}$ C on bitumen represented in Black space:

(A) equivalent complex shear modulus evolution during each loading period, measured at 2 % shear-strain amplitude and (B) complex modulus evolution, during each rest period of 4 h, measured at 0.05 % shear-strain amplitude (from Babadopulos et al.<sup>11</sup>).



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can be considered as a constant while the norm of complex modulus decreases after each loop. The evolution 140 during loading periods is because of damage but also thixotropy. After correcting for damage, the results from the 141 Black Space diagram show the different directions of nonlinearity and thixotropy. 142

# Rheological Assessment of Cracking Distress

In addition to the use of Black Space to assess asphalt material damage and healing and other physical phenomena, it is also possible to use these diagrams to identify critical limits associated with different forms of material cracking. Various rheological parameters can be defined and graphically displayed in Black space to allow critical and failure regions of cracking response to be identified and material changes with regard to aging and rejuvenation to be mapped.

#### THERMAL CRACKING - BENDING BEAM RHEOMETER DATA BLACK SPACE

The development of the *S* and *m* criteria for thermal cracking was validated by inspection of built pavements by 150 Leahy, Harrigan, and Von Quintus.<sup>12</sup> Subsequently, Rowe<sup>13</sup> showed how the *S* and *m* criteria could then be 151 translated to  $|G^*|$  and phase angle using a transformation method by fitting a discrete spectrum to the bending 152 beam rheometer (BBR) data and performing an interconversion to the dynamic properties. 153

Once the data are transformed, it is then possible to take the criteria and insert these into Black Space where C11 the limiting criteria become a value of  $|G^*|$  (111 MPa) and phase lag,  $\delta$  (26.2°) at a frequency of 0.0167 radians/s 155 (1/60 s), that correspond to the *S* and *m* values (at 60 s) as shown in **figure 4**. Thus, we observe that the *S* and *m* 156 criteria are essentially the same as the specification of a modulus and phase angle in the Black space. 157

#### **DURABILITY CRACKING - GLOVER-ROWE PARAMETER**

One approach to assess the cracking resistance of asphalt materials, particularly durability cracking, is through the 159 use of Black Space diagrams and a point parameter known as the Glover-Rowe (*G-R*) parameter. Based initially on 160 the relationship between ductility and pavement performance and an understanding of the importance of the 161 stress relaxation properties of the binder in an asphalt mixture, the *G-R* parameter can be used to evaluate the 162 resistance of binder and asphalt mixtures to age-related cracking.<sup>14–16</sup> The *G-R* parameter for binders can 163 be computed from measurements of complex modulus ( $|G^*|$ ) and phase angle ( $\delta$ ) using a DSR at a frequency 164 of 0.005 rad/s and 15°C using equation (1).

$$G - R = |G^*| (\cos \delta)^2 / \sin \delta \tag{1}$$

By plotting the *G-R* parameter and including the limiting values associated with a warning of the probability 166 of cracking (180 kPa) and presence of significant cracking (600 kPa) (values derived from field durability cracking 167 evaluation<sup>14</sup>), it is possible to identify the effect of aging on the susceptibility of a binder to durability cracking as shown in figure 5.

Similarly, the same concept can also be considered for rejuvenation as shown in **figure 6** for two locations 170 treated with a rejuvenator.<sup>17</sup>

**Figure 6** also shows lines that represent the rheological index (*R*-value) as defined by the Christensen-172 Anderson (CA) model<sup>18</sup> with a glassy modulus ( $G_g$ ) set as 1 GPa. However, this assumption that the value 173 of  $G_g$  is equal to 1 GPa for all binders is most likely incorrect. Other works have shown that this value can vary 174 considerably and also changes with temperature susceptibility of the binder.<sup>19</sup> Thus, the assumption that R = 175 9-log  $G_c$ , where  $G_c$  is the crossover modulus is incorrect, and it has been suggested that it is better to simply use the value of  $G_c$  as a specification parameter rather than R.  $G_c$  has the advantage in that it lies in a similar stiffness range to the *G-R* value for most paving binders and effectively characterizes the shape of the master curve in the high stiffness region whereas the *G-R* value identifies the hardness of the binder. Other parameters being considered for specifications in the intermediate temperature/non-load related cracking region also include the phase angle 180





#### FIG. 5

Black Space diagram including *G-R* parameter limits. Different binders at different stages of aging.<sup>17</sup>



#### FIG. 6

Black Space diagram including *G-R* parameter limits. Treatment of binders with rejuvenators (other rheological indices,  $\Delta T_c$ and *R*-values also shown).<sup>17</sup>



response at a stiffness of 8,967 kPa,  $\delta_{G^*=8.967 \text{ GPa}}$ .<sup>20</sup> It should be noted that this parameter, the *R*-value, the *G<sub>c</sub>* and 181  $\Delta T_c$  (shape parameter determined as the difference between the critical temperatures of  $T_{stiffness}$  (60 s) and  $T_{m-value}$  182 (60 s) from the BBR) all define the shape of the master curve in the intermediate temperature region, much in the 183 same manner that the penetration index or penetration-viscosity number would characterize properties using 184 empirical specification approaches. 185

By inspection of the data in **figure 6**, we note that at an *R*-value of 2, the value of  $|G^*|$  is close to a value of 186 1 MPa at the critical region where the *G*-*R* parameter transitions from a "cracking warning" condition to a "cracking limit" condition. Several approaches exist for understanding the stiffness and relation properties in this stiffness range. Regardless, we note that it is important that both stiffness and relation properties are defined 189 in this region. The advantage of the *G*-*R* parameter to other parameters is that as materials age, the relationship produced intersects with the *G*-*R* lines at nearly a right angle, indicating that the parameter is very sensitive to the changes in the properties that are occurring because of aging.

As discussed earlier, the rheological index (*R*-value) as defined by the CA model,<sup>18</sup>  $\delta_{G^*=8.967 \text{ GPa}}$ ,  $G_c$ , and  $\Delta T_c$  193 effectively define the shape of the master curve in the higher stiffness region ( $|G^*| \ge 10^5$  Pa). The selection 194 of a parameter for specification use should depend upon the ease of measurement (likely to significantly affect 195 implementation) and the accuracy of testing as determined by interlaboratory studies (precision, repeatability, 196 reproducibility, etc.). However, while the shape of the master curve is of considerable importance, the relative 197 hardness (stiffness) is needed. This can be captured by the *G-R* parameter, which can be regarded as a "point" 198 parameter. Other parameters that effectively capture the hardness of the material include the crossover frequency, 199  $\omega_{cr}$  (reference temperature must be specified) or the temperature at which the crossover modulus occurs,  $T_{cr}$  or 200  $T_{VET}$ , (frequency must be specified). However, these last two parameters cannot be easily plotted in Black Space, 201 and the conceptual tie with performance is more difficult to visualize. 202

#### DURABILITY AND THERMAL CRACKING

As discussed earlier, a direct relationship exists between measurements conducted in the frequency and time 204 domain provided that strains are kept within the LVE region. For asphalt binders, we have made use of inter-205 conversion technologies using the RHEA software and the computation of the calculated relaxation spectra fit to 206

the data. The parameters for limiting cold-temperature thermal cracking (S = 300 MPa and m = 0.300) thus can 207 be translated into values of  $|G^*| = 111$  MPa and  $\delta = 26.8^\circ$  at a frequency of 0.0167 radians/s (1/60 s). This intercept of complex modulus and phase lag can be shown as a *G*-*R* relationship that has a value of 184 MPa, and 209 this could be used as an alternate to the *S* and *m* values currently used as shown together with other Black Space 210 parameters in figure 7. 211

In earlier work, Anderson et al.<sup>16</sup> showed that a good correlation between  $\Delta Tc$  and the *G-R* parameter existed. However, because one of the parameters is a shape parameter whereas the other is a point parameter, no correlation should be expected across the wide range of materials used in asphalt paving. Rowe<sup>21</sup> presented data that demonstrated that binders with different rheological types have significantly different behavior when comparing the *G-R* parameters to  $\Delta Tc$ . Specifically, elastomeric binders that are known to perform better with respect to cracking often have low values of  $\Delta Tc$ .

Zhang, Sias, and Dave<sup>22</sup> presented similar data in the format of *G-R* versus  $\Delta Tc$  that shows the relationships for different binders with *G-R* and  $\Delta T_c$  parameters as shown in **figure 8**. In this study, the propensity to 219 cracking of reclaimed asphalt pavement (RAP)-modified materials used in the northeastern USA was studied **C12** with binders after different aging conditions. The dashed lines represent the cracking-warning values, and 221 the solid lines represent the cracking-limit values for the two parameters. Points in the bottom right quadrant 222 of the plot are acceptable under both criteria whereas those in the top left quadrant would be sensitive to 223 cracking using both criteria. The short-term conditioned binders (first point on each line) generally fall into 224 the safe zone, which means that typically no cracking problems are expected. As the materials age, they move 225 toward the upper left quadrant, indicating that there may be significant cracking problems based on the  $\Delta T_c$  226 and *G-R* criteria.

#### FATIGUE CRACKING AND CRACKING PERFORMANCE LIMITS

In addition to representing the *G-R* parameter in Black Space and providing limits for the onset and failure 229 associated with durability cracking, it is also possible to represent other non-load and load-related cracking 230

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#### FIG. 7 Black Space representation of various parameters used to define cracking and deformation for binders.<sup>17</sup>





and rutting limits. The Black Space diagram can depict pass and fail limits for rutting as well as forms of pavement 231 cracking (durability, fatigue, and thermal) as shown earlier. It should be noted that at the stiffness values con-232 sistent with rutting, the binder is generally behaving in a different manner. For example, we know that as the 233 stiffness drops below  $10^5$  Pa that the CA model loses its validity.<sup>18</sup> 234

For conventional binders,  $\Delta Tc$  is strongly correlated with the *R*-value. Specifiers generally consider limits for 235  $\Delta Tc$  in specifications with values around -3.5 to -5. Values lower than this would represent binders that are cluded from specifications. The *R*-value of 1.92 corresponds approximately to a  $\Delta Tc$  value of 0. This value can 237 vary to a small extent with changes in the glassy modulus and temperature susceptibility. An *R*-value of 3.0 is a 238 practical limit related to a low (negative) value of  $\Delta Tc$  of around -5.0. This value of *R* is typical of an oxidized or 239 aged binder and represents a temperature susceptibility defined by  $\Delta T_c$  in the critical range.<sup>17</sup> A low *R*-value 240 represents a binder that is not practical because the material will generally have a high temperature susceptibility. 241 Thus, conventional asphalt binders will lie within the shaded area as shown in figure 9. 242

The specification of an *R*-value is problematic because this parameter is not easily determined and is a 243 function of the analysis method.<sup>17</sup> However, if a constant value of 1 GPa is assumed for the glassy modulus, 244 the control of the *R*-value can be directly correlated to the crossover modulus,  $G_{c}$  (the value of  $|G^*|$  at a phase 245 angle of 45°), which can be easily measured for paving grade binders. This effectively allows both  $\Delta Tc$  and G-R to 246 be controlled by Black Space parameters. 247

Thus, in summary for binders, the critical binder parameters associated with behavior in a Black Space plot, 248 including those developed under the SHRP program, become the following: 249

- Thermal: G-R, 184 MPa measured at a slow frequency (1/60 s) 0.0167 rads/s at  $T_{min}$  + 10C or  $-|G^*| < \frac{AQZ}{AQS}$ 111 MPa and  $\delta > 26.2^{\circ}$  at the same conditions.
- Fatigue (SHRP): |G\*|sinδ measured at 10 rads/s at intermediate temperature parameter as developed by 252 SHRP researchers, which is under review.
- Durability: *G-R* parameter measured at 15°C and 0.005 rads/s with two criteria being considered (180 kPa 254 and 600 kPa) and Gc > 1 MPa, which effectively removes the need to specify  $\Delta Tc$ . 255



**FIG. 9** Extending Black Space parameters with limits associated with ranges in  $\Delta Tc$ .

Deformation: |G\*| / sinδ (1/J<sup>\*</sup>) as currently used – but now because of nonlinear effects, it is proposed that 256 the multiple stress creep recovery test will be a better test

#### **G-R** CONCEPT FOR ASPHALT MIXTURES

Based on the binder *G-R* approach, Mensching, Rowe, and Daniel<sup>23</sup> developed the mixture Glover-Rowe (*G-R<sub>m</sub>*) 259 parameter using stiffness and phase angle measured on the asphalt mixture ( $|E^*|$  and  $\delta$ ). However, determining a 260 suitable temperature and frequency to calculate *G-R<sub>m</sub>* has required considerable research effort. Oshone et al.<sup>24</sup> 261 used results from 81 asphalt mixtures to assess different temperature and frequency combinations (**fig. 10**). This 262 work observed that most discrimination between mixtures in terms of  $|E^*|$  and phase angle was observed at 15°C 263 and 5rad/s and PGLT + 10 and 0.01666rad/s (where PGLT is the low-temperature performance grade parameter). AQ9 Subsequent research has further developed *G-R<sub>m</sub>* to use a commonly measured temperature-frequency combination of 20°C and 5 Hz.<sup>25,26</sup> 266

Oshone et al.<sup>24</sup> further conducted Pearson's correlation analysis on the  $G-R_m$  parameter (15°C and 5 rad/s) 267 from the 81 asphalt mixtures. As expected, the binder content and voids in mineral aggregate showed strong 268 correlations where an increase in these parameters results in lowering of  $G-R_m$ . PGLT also showed a strong 269 correlation where with lowering of PGLT, the  $G-R_m$  reduces (in other words, a better PGLT lowers the value 270 of  $G-R_m$ ). Increasing nominal maximum aggregate size for mixtures increases  $G-R_m$ , and increasing amounts of 271 RAP as well as PG high-temperature grade increases  $G-R_m$  but with a smaller degree of correlation. 272

 $G-R_m$  can be used to compare various mixtures (binder grades, recycled material content, etc.) and to evaluate and quantify changes in viscoelastic material properties with aging (e.g., fig. 11) or with rejuvenation. Kaseer et al.<sup>27</sup> used mixture Black Space and the  $G-R_m$  parameter to evaluate various strategies for producing mixtures with higher RAP contents. Various recycling agents and use of a softer virgin binder were also evaluated at both short- and long-term aging conditions. Zhang, Sias, and Dave<sup>22</sup> used the  $G-R_m$  to correlate laboratory conditioning methods with field-aging durations for a set of mixtures. Zhang<sup>28</sup> also developed a rheology-based mixture 278

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#### FIG. 10

Discrimination between mixtures in terms of  $|E^*|$ and phase angle at different frequency and temperature combinations (reproduced using data from Oshone et al.<sup>24</sup>).



FIG. 11 Mixture G-R parameter evolution with aging (from Ogbo et al.<sup>26</sup>).



aging model using the G- $R_m$  parameter to evaluate cracking and aging susceptibility of mixtures with different 279 binder grades and RAP content. 280

Master curve shape parameters obtained from fitting mixture  $|E^*|$  and phase angle master curves are also 281 used to track and quantify the impact of aging and rejuvenation on the mixture viscoelastic properties.<sup>29</sup> 282

#### EVALUATING RECYCLED MATERIALS, AGING, AND REJUVENATION

Recycled asphalt content in asphalt binders and mixtures have prominent effects on their rheological properties. 284 A study by Daniel et al.<sup>30</sup> extensively evaluated effects of RAP content on Black Space parameters. An example 285 result from that research is presented in **figure 12** and shows the Black Space diagram (along with *G-R* parameter 286 limits) for extracted and recovered binders from plant-produced mixtures as a function of RAP content (0, 20, 287 and 40 % by total weight of mix) and binder amount (optimum, optimum -0.5 %, and optimum +0.5 %). Results 288 for tank binder used in production are also shown in the figure for comparison. The results show that increasing 289

FIG. 12 Black Space diagram for asphalt binders extracted and recovered from asphalt mixtures with varying RAP amounts and binder contents (produced using data from Daniel et al.<sup>30</sup>).



the RAP amount and decreasing binder content led to significant increases in binder stiffness and loss of phase 290 angle with higher RAP mixtures (40 % RAP) all in the "crack warning" region for the G-R parameter and the low-291 asphalt content mixture with high RAP amount past the cracking limit. The effect of lowering binder content on 292 the G-R parameter can be attributed to decreases in asphalt binder film thickness and thus increased aging during 293 mix production. 294

Data from the same study by Daniel et al.<sup>30</sup> also include assessment of *R*-value and crossover frequency for 295 the binders extracted from asphalt mixtures with varying RAP amounts and binder contents. As expected, introduction of RAP in the mixture results in a significant reduction in crossover frequency. While RAP amount 297 increases from 20 to 40 %, the *R*-value increases, with this effect being more pronounced for mixtures with a lower 298 binder content. 299

Black Space and the *G-R* parameter can also be used to access both the aging and rejuvenating effects linked 300 with binders associated with recycled asphalt pavements. Rahbar-Rastegar, Daniel, and Dave<sup>31</sup> used mixture 301 Black Space to compare various laboratory conditioning protocols for mixtures and to evaluate how both 302  $|E^*|$  and phase angle master curves change with aging. 303

Figure 13 shows an example of the evolution of the G-R parameter (measured on extracted and recovered 304binders) with mixture-aging conditions. In this study, Zhang, Sias, and Dave<sup>22</sup> used this analysis to determine the 305mixture-aging condition that corresponds to the standard 20-h pressure aging vessel aging on the virgin binderand also to evaluate the change in aging with depth using measurements from field cores.307

The extent of virgin and RAP binder blending is presented in figure 14 with short-term and long-term 308 aging conditions (indicated by the rolling thin-film oven test and pressure aging vessel).<sup>32</sup> The Black C16 Space diagram shows the effect of different degrees of binder activation, as a measure of virgin and RAP binder 310 blending, and the shift toward the top left-hand corner with aging and in the opposite direction with 311 rejuvenation. 312

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FIG. 13 Evolution of G-R parameter with aging for virgin and extracted and recovered binders.<sup>22</sup>

FIG. 14 G-R parameter for recycled binders showing aging and rejuvenating effects.<sup>32</sup>



The effect of different types of recycling agents can also be compared using Black Space and the *G-R* parameter. Work by Bajaj et al.<sup>33</sup> showed how different types or categories of recycling agents produce primarily softening effects (reduction in  $|G^*|$  only) or rejuvenating effects (reduction in  $|G^*|$  and increase in phase angle). This study also evaluated the impact of different conditioning sequences on the rheological parameters of binders blended with RAP and recycling agents to examine how the interactions between the different materials impact rheological changes with aging.

## Rheological Assessment of Alternative Binders

The techniques and approaches that have made use of Black Space have generally been applied to conventional 320 asphalt materials. However, the move toward more sustainable solutions to minimize environmental impact has 321 resulted in increased use of industrial byproducts and wastes in asphalt mixtures with the idea of partially replac-322 ing asphalt binders with bio-based materials or alternative binders. These synthetic binders or bio-based materials 323 (also known as bio-binders or bio-oils) can be considered as renewable materials as compared to conventional 324 asphalt binders that are derived from fossil fuels.<sup>34–36</sup> 325

FIG. 15 Black Space diagram of rheological data for different conventional (70/100 pen), polymer-modified (SBS PMB), tire rubber-modified (TRN MB), and synthetic binders (MA, EA) (after Airey et al.<sup>44</sup>).



Most of these renewable bio-based materials are of vegetative origin such as lignin, tall oil, pitch, wood resins, 326 and plant sap.<sup>35</sup> Another widely used renewable material of vegetative origin is waste cooking oil, which is available in 327 large quantities all over the world.<sup>37,38</sup> Further examples are bio-oils obtained from vegetable oils and biomass such as 328 soy, linseed, and rapeseed. Other sources of bio-binders include algae,<sup>39</sup> wood residues,<sup>40–42</sup> and swine manure.<sup>43</sup> 329

Another aspect to be considered is the complexity of the treatment before the employment of these renewable materials. Most bio-based materials used for asphalt materials are obtained from raw materials through 331 thermal treatments, chemical treatments, or both, such as pyrolysis, esterification, and hydrothermal liquefaction. 332 These production techniques add an extra dimension to the rheological complexity of the final binder. 333

Black Space diagrams can be considered to provide rheological "fingerprints" for these different binders and 334 have been used in **figure 15** to provide a convenient method of comparing different categories of binders such as 335 standard paving grade binders, modified binders, and synthetic polyacrylate binders.<sup>34,44</sup> The Black Space curves 336 allow the unique rheological behavior of the polyacrylate binders to be illustrated without the need for more 337 advanced rheological characterization involved with the generation of master curves. 338

Black Space diagrams have also been used for bio-binders produced using lignin and sawdust and produced 339 using pyrolysis and hydrothermal liquefaction procedures at different operating conditions. Similar to the rheological behavior shown in figure 15, the rheological Black Space curves can be used to show the unique viscoelastic 341 properties of these bio-based materials. 342

# **Concluding Remarks**

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The paper has described three broad applications of the use of Black Space diagrams in terms of the rheological 344 assessment of asphalt binders and mixtures to aid interpretation and analysis of material and pavement perfor-345 mance. These three applications included global damage and healing assessment, rheological characterization for 346 cracking behavior evaluation (including effects of recycled materials, aging, and rejuvenation), and rheological 347 fingerprinting of alternative synthetic or bio-based sustainable asphalt binders. 348

In terms of cyclic loading and fatigue tests, Black Space diagrams have been used to evaluate the damage and 349 healing changes in complex modulus and phase angle through the selection of strain amplitude sweep tests as well 350 as combination loading and rest period cyclic tests. The results from these Black Space diagrams have enabled 351 nonlinearity as well as the phenomena of self-heating and thixotrophy to be identified in addition to changes in 352 complex modulus and phase angle because of damage and healing. 353

In terms of low-temperature (thermal) cracking, it is possible to transform the Superpave S (300 MPa) and 354 m (0.3) criteria to a limited complex modulus (111 MPa) and phase angle (26.2°) set of criteria at a frequency of 355 0.0167 radians/s with these limits being used in Black Space to identify regions of high or low thermal-cracking 356 susceptibility.

Black Space diagrams and a point parameter known as the *G-R* parameter have been successfully used to 358 assess the durability (age-related) cracking resistance of asphalt materials. The *G-R* parameter, together with 359 limiting values associated with a warning of the probability of cracking (180 kPa) and presence of significant 360 cracking (600 kPa), can be presented in Black Space to monitor the susceptibility of asphalt binders to aging 361 as well as the reverse effect of rejuvenation. 362

With the ability to transform the thermal cracking criteria of *S* and *m* to  $G^*$  and  $\delta$ , it is also possible to link 363 thermal cracking with durability cracking by defining a *G*-*R* value of 184 MPa for thermal cracking. Although not 364 a direct correlation, it is also possible to link the *G*-*R* parameter and  $\Delta Tc$  to identify asphalt materials that have 365 a greater or lower potential for significant cracking. 366

In addition to durability cracking, the Black Space diagram can also depict pass and fail limits for rutting 367 as well as various forms of non-load and load-relating pavement cracking. It is also possible to establish relationships between  $\Delta Tc$ , *R*-value, and the *G*-*R* parameter and provide a shaded area in the Black Space diagram for conventional asphalt binders. 370

It is also possible to extend the *G*-*R* parameter to asphalt mixtures (G- $R_m$ ) with most measurements made at 371 a temperature-frequency combination of 20°C and 5 Hz. The *G*- $R_m$  parameter can then be used to evaluate 372 and quantify changes in viscoelastic material properties with aging or with rejuvenation. Black Space and the 373 *G*- $R_m$  parameter are also used to assess the cracking and aging susceptibility of asphalt mixtures with different 374 contents and types of RAP. 375

Finally, Black Space has been used to illustrate the complex rheological response of both synthetic binders 376 and bio-binders with some of these binders showing useful combinations of high stiffness as well as good 377 relaxation capabilities. 378

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