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Quantitative relationship of fundamental rheological properties of bitumen with the empirical Ring and Ball softening point

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\textbf{ABSTRACT}

This paper employs the linear viscoelastic parameter $G'/\tan\delta$ as the quantitative criterion for identifying a common relationship with the Ring and Ball softening point of both unmodified and polymer-modified bitumen (PMB). Temperature sweep was conducted at 10 rad/s with dynamic shear rheometer to determine the complex shear modulus $G^*$ and phase angle $\delta$ of bitumen. The parameter $G'/\tan\delta$ could be obtained. Logarithmic interpolation was applied for $G'/\tan\delta$ to determine the equivalent softening temperature $T_{s,G'/\tan\delta}$ at which $G'/\tan\delta$ equals the proposed criterion values. With this method, a good agreement could be reached between $T_{s,G'/\tan\delta}$ and the softening point for both unmodified bitumen and PMB. While keeping high accuracy for unmodified bitumen, the accuracy of $T_{s,G'/\tan\delta}$ as an estimation of the softening point of PMB was largely improved when compared to the iso-modulus approach. The reliability of this quantitative relationship is preliminarily verified and its implementation is discussed in the Black space.

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\textbf{KEYWORDS}

Bitumen; softening point; dynamic shear rheometer; temperature sweep; rheological property

1. Introduction

Currently, the penetration-based specification for paving bitumen is still in use in many countries all over the world, e.g. European countries, China, etc. In penetration-based specifications, the softening point is one of the most basic technical parameters for bitumen. It is usually measured with the empirical Ring and Ball method, which is standardised in EN 1427, ASTM D36, etc. As in EN 1427, a steel ball of 3.5 g weight and 9.5 mm in diameter is put on the disc-shaped bitumen sample that is cast in a brass ring and immersed in a liquid bath. The bath is heated up at a rate of 5°C/min and the softening point is determined as the temperature at which the bitumen softens enough to allow the ball to fall a distance of 25 mm. Water is used as the bath liquid for the softening point determination below 80°C while glycerol is used for the measurement above 80°C. According to EN 1427, there is an approximately 4°C difference when a given bitumen is tested in the different baths (usually higher in the glycerol bath than in the water bath).

Softening point is very often the only technical parameter in a penetration-based specification to regulate the high-temperature performance of bitumen. As a performance indicator, it works well for unmodified bitumen but can be problematic when it comes to polymer-modified bitumen, i.e. PMB (BiTVal, 2006; FunDBitS, 2016; Jelagin et al., 2018). Towards performance-related specifications, the dynamic shear rheometer (DSR) has been increasingly used to test and characterise bitumen (Eurobitume, 2009, 2012; Southern, 2015). As the development of new analysis methods and technical

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parameters with DSR, a slow and gradual transition from the penetration-based bitumen specification to performance-related specification using DSR is foreseeable in many countries in the near future, especially for PMB.

During this transition, it is of great importance to understand the relationship between the old and new specifications. In some cases, it can even be necessary to understand the quantitative relationship. This helps in the formulation and revision of the new specification based on past experience as well as in building up new experience securely. Additionally, this also enables the estimation of the old empirical parameters from fundamental DSR results, which would be more efficient and timesaving than running extra tests. Furthermore, a reliable quantitative relationship can in turn assist in understanding the reasons why the old empirical parameters were functioning or problematic. From this perspective, the present paper investigates the quantitative relationship of DSR results with the softening point of both unmodified and modified bituminous binders. It should be noted that searching for more performance-related bitumen parameters is not in the scope of this paper.

In the history, it was found that most conventional bituminous binders (i.e. unmodified) have a penetration value of 800 dmm and a viscosity of about 1300 Pa·s at the softening point (Heukelom, 1973; Koenders, 2015; Pfeiffer & Van Doormaal, 1936). More recently, some previous studies investigated the softening of bitumen and attempted identifying the equivalent softening temperature of bitumen based on DSR results. Nigen-Chaidron (2008) reported the concept of ‘equi-viscous temperature’ (EVT), defined as the temperature at which the complex shear viscosity of bitumen is equal to 2 kPa·s. Zoorob et al. (2012) employed this approach and demonstrated that the EVT estimates the softening point of unmodified bitumen very well but does not function for PMB. Fan et al. (2014) proposed the concept of ‘equivalent modulus temperature’, at which the complex shear modulus of bitumen is equal to 13 kPa at 10 rad/s in the linear viscoelastic range. It was concluded that this temperature is a rather good estimation of the softening point for the studied unmodified binders, but not for the PMB. This iso-modulus approach was also adopted by Alisov et al. (2020) to develop a new bitumen evaluation system with the BTSV method (Bitumen-Typisierungs-Schnell-Verfahren in German, meaning Binder-Fast-Characterisation-Test in English). The BTSV method is based on the complex shear modulus of bitumen equal to 15 kPa at 10 rad/s. The equivalent temperature $T_{BTSV}$ is a good estimation of the softening point for unmodified bitumen, however, not for PMB.

Neither the EVT nor the iso-modulus approach considers the role of phase angle in the softening of bitumen, which might be the reason why these approaches do not lead to accurate estimations for the softening point of PMB. Lu and Isacsson (1997a, 1997b) investigated dozens of modified bituminous binders with various polymer modifiers and found a good correlation between the softening point and the temperature at which the phase angle is equal to 75° at 1 rad/s. This correlation indicates that the phase angle is crucial for quantitatively relating the rheological properties to the softening point of PMB. Towards a common quantitative relationship for both unmodified and modified binders, both the complex shear modulus $G^*$ and phase angle $\delta$ should be considered.

For this, Kriz et al. (2016) investigated hundreds of bituminous binders in two databases and reported the correlation between softening point and the equivalent temperature at which the parameter $G^*/\sin \delta$ is equal to 11,572 Pa. It was found that this equivalent temperature correlates rather well with the softening point for unmodified binders, but the correlation was poor for PMB. As Zoorob et al. (2012) suggested, the factor $\sin \delta$ is very insensitive to phase angles greater than 60°. Thus, the parameter $G^*/\sin \delta$ does not make a very big difference from the $G^*$, i.e. the iso-modulus approach, regarding bitumen properties around the softening point. However, further investigations on other rheological parameters that consider both $G^*$ and $\delta$ are still necessary and promising towards a common quantitative relationship with the softening point of both unmodified and modified bituminous binders. The potential criterion parameters should be linear parameters but are able to indicate the non-linear behaviour of bitumen to some extent. This paper reports the searching for such a quantitative relationship and the evaluation of its reliability with both unmodified and modified bitumen.
2. Materials and method

2.1. Materials

Five bituminous binders were analysed in this study, including three unmodified (penetration grades 50/70, 70/100, and 160/220) and two modified binders with the same type of linear styrene–butadiene-styrene (SBS) copolymer. The SBS-modified binders were prepared in the laboratory by mixing the SBS modifier (powder) with a base bitumen of penetration grade 100/150. The preparation was carried out at 180°C and the mixing process lasted for 3 h. The polymer contents of the two modified binders were 3% and 5% by weight of the blends. Their needle penetration values were respectively 93 and 71 dmm at 25°C according to the standard EN 1426 (with 100 g load and 5 s time). These two modified binders are denoted as 'PMB 3% SBS+100/150' and 'PMB 5% SBS+100/150' in this paper while the unmodified binders are denoted with their penetration grades. Both the original binders and short-term aged binders were investigated in this study. The short-term ageing was done in the laboratory with the rolling thin film oven test (RTFOT) according to the standard EN 12607-1 (at 163°C for 75 min).

2.2. Method

The softening point of bitumen was measured with the Ring and Ball method according to the standard EN 1427. DSR temperature sweep was done according to the standard EN 14770 at 10 rad/s between 4 and 82°C with a 6°C interval. The plate-plate geometry was used for the DSR measurement. At higher temperatures, the plates of 25 mm diameter were used with 1 mm gap. At lower temperatures, the plates of 8 mm diameter were used with 2 mm gap. The change of plates was done around the \( G^\ast \) level of 100 kPa. At the temperature of plate change, both plate sizes were used to compare the results and check the repeatability of the measurement. For analysing the quantitative relationship with the softening point, the iso-modulus approach was taken as the reference method. All the binders were analysed with the increasingly popular BTSV method and the \( T_{\text{BTSV}} \) values were obtained.

Towards a common quantitative relationship with the softening point of both unmodified and modified binders, the searching for potential criterion parameters started with reviewing the reported rheological data in the literature. According to previous studies (Alisov et al., 2020; Fan et al., 2014), PMB usually shows lower \( G^\ast \) and lower \( \delta \) values than unmodified bitumen at the softening point. A simple mathematical analysis disqualifies the viscous shear modulus \( G^{''} \) (i.e. \( G^\ast \cdot \sin \delta \)) as a potential criterion parameter while the elastic shear modulus \( G^{'} \) (i.e. \( G^\ast \cdot \cos \delta \)) is preliminarily qualified. Another parameter, \( G^{’}/\tan \delta \), has even greater potential as a candidate criterion parameter, due to the possible link to the non-linear behaviour of bitumen.

The parameter \( G^{’}/\tan \delta \) is usually used for the Glover-Rowe parameter. It can be rewritten in another form as \( G^\ast \cdot (\cos \delta)^2/\sin \delta \). This parameter was initially derived in its original form as \( G^{’}/(\eta^{’}/G^\ast) \) by Glover et al. (2005) based on the Maxwell model, indicating the balance between the stiffness and the viscous flow or ductile behaviour of bitumen (Garcia Cucalon et al., 2019). It was found that this parameter at 15°C and 0.005 rad/s, which can be equivalently shifted to 44.7°C and 10 rad/s according to the time-temperature superposition principle, correlates well with the ductility of bitumen at 15°C and 1 cm/min. In this case, the linear parameter serves as a link to the non-linear behaviour of bitumen. After the further derivation by Rowe (2011, 2014), the Glover-Rowe parameter has been mostly used as a stand-alone parameter for predicting the fatigue and non-load associated cracking susceptibility of binders at intermediate temperatures (Anderson et al., 2011; Mogawer et al., 2017; Sharma et al., 2017). However, its use at 10 rad/s and higher temperatures has been rarely reported. This paper investigates the possibility of quantitatively relating this parameter to the softening point of bitumen.

By interpolating between the DSR results, the \( G^\ast \) and \( \delta \) values of the studied binders at their respective Ring and Ball softening point temperatures were obtained. Logarithmic interpolation was employed for the modulus while linear interpolation was employed for the phase angle and temperature. Then, the values of the qualified candidate criterion parameters were calculated and analysed.
at the Ring and Ball softening point, including $G', G'/\tan\delta$ and the previously reported $G*/\sin\delta$. Based on this analysis, a quantitative relationship with the softening point was identified and investigated for reliability. Besides the above-mentioned binders in this paper, more binder data were obtained by searching in the literature to preliminarily verify the reliability of the identified relationship.

3. Results and discussion

3.1. Bitumen testing

The Ring and Ball softening point results of the studied binders are presented in Table 1. All the binders had the softening point below 80°C, tested in the water bath. The unmodified bitumen had an increase by about 5°C in softening point after RTFOT ageing while the SBS-modified binders increased by about 1°C. The DSR temperature sweep results at 10 rad/s are shown in Figures 1 and 2, respectively for the original and RTFOT-aged binders. The result comparison at the temperature of plate change indicates good repeatability of the measurement.

For the unmodified binders, it can be seen in Figures 1 and 2 that a lower penetration grade (harder bitumen) reveals higher $G*$ and lower $\delta$ values in the entire investigated temperature range. The

Table 1. Softening point results of bitumen by the Ring and Ball method (EN 1427).

<table>
<thead>
<tr>
<th>Property</th>
<th>Softening point, original (°C)</th>
<th>Softening point, after RTFOT (°C)</th>
<th>Softening point change (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bitumen 50/70</td>
<td>50.8</td>
<td>56.0</td>
<td>5.2</td>
</tr>
<tr>
<td>Bitumen 70/100</td>
<td>46.6</td>
<td>51.6</td>
<td>5.0</td>
</tr>
<tr>
<td>Bitumen 160/220</td>
<td>38.0</td>
<td>43.2</td>
<td>5.2</td>
</tr>
<tr>
<td>PMB 3% SBS + 100/150</td>
<td>51.2</td>
<td>52.4</td>
<td>1.2</td>
</tr>
<tr>
<td>PMB 5% SBS + 100/150</td>
<td>75.4</td>
<td>76.4</td>
<td>1.0</td>
</tr>
</tbody>
</table>

Figure 1. DSR temperature sweep (10 rad/s) results of original bitumen.
SBS-modified binders followed largely the $G^*$ trend of the base bitumen at lower temperatures, i.e. between penetration grades 70/100 and 160/220 with possible ageing effects during the PMB preparation. However, the modification with SBS copolymer did lift the $G^*$ up at higher temperatures. In addition, the PMB binders showed decreased $\delta$ values, especially at higher temperatures. When the SBS content is higher in the PMB, the lifting effect on $G^*$ and the reduction effect on $\delta$ become more significant.

3.2. The BTSV analysis

Based on the DSR temperature sweep results presented above, the BTSV method (Alisov et al., 2020) was employed to analyse the binders. Logarithmic interpolation was applied for $G^*$ to determine the equivalent temperature $T_{BTSV}$ at which $G^*$ is equal to 15 kPa at 10 rad/s (linear interpolation for temperature). The corresponding $\delta_{BTSV}$ (phase angle at $T_{BTSV}$) was determined as well by linear interpolation. As an example, the determination for original Bitumen 160/220 is presented in Figure 3. All the other binders were analysed in the same way and the $T_{BTSV}$ and $\delta_{BTSV}$ values for each binder were obtained.

In Figure 4, the obtained $T_{BTSV}$ value of each binder is plotted against the Ring and Ball softening point. As expected, the results show that the equivalent temperature $T_{BTSV}$ is a rather good estimation of the softening point for the unmodified binders, both before and after the RTFOT ageing. Even for the modified bitumen with 3% SBS copolymer, the $T_{BTSV}$ was quite close to the measured softening point. However, for the modified bitumen with 5% SBS, the difference between $T_{BTSV}$ and the measured softening point was large, i.e. a more than 20°C difference. This is due to the fact that PMB usually shows a lower $G^*$ value than unmodified bitumen at the softening point (Alisov et al., 2020; Fan et al., 2014). When the $G^*$ level for unmodified bitumen is used to determine the equivalent temperature, this temperature tends to underestimate the softening point of PMB. This does not necessarily mean that $T_{BTSV}$ is less meaningful for characterising PMB but could mean the opposite. The correlation of
these test methods, both the BTSV and Ring and Ball softening point, with the bitumen performance in the pavement is another important research question that needs to be discussed but is not in the scope of this study. This study focuses on the relationship between different binder test methods.

The BTSV method takes the iso-modulus approach ($G^*$ equal to 15 kPa at 10 rad/s) and creates a new system for bitumen evaluation based on the equivalent temperature $T_{BTSV}$ and corresponding phase angle $\delta_{BTSV}$, i.e., the $\delta_{BTSV}$-$T_{BTSV}$ plot. Figure 5 shows the obtained results of the studied binders in such a plot. For the original unmodified binders, it can be seen that a lower penetration grade (harder bitumen) reveals a higher $T_{BTSV}$ value while the $\delta_{BTSV}$ value does not vary very much (all about 82°).

When the unmodified binder was aged by the RTFOT method, its $T_{BTSV}$ value became higher and $\delta_{BTSV}$ value became lower (all to about 80°). According to Alisov et al. (2020), this trend would continue if the ageing continued. The modified bitumen with 3% SBS followed the same trend in terms of ageing, but with already lowered $\delta_{BTSV}$ values as compared with the unmodified binders. The modified bitumen with 5% SBS showed an even lower $\delta_{BTSV}$ (and increased $T_{BTSV}$ with more SBS) and kept the $\delta_{BTSV}$...
value almost unchanged after the RTFOT ageing. Overall, the BTSV method leads to an iso-modulus evaluation of bitumen at the modulus level of unmodified bitumen at the Ring and Ball softening point. However, this level is usually much higher than the modulus of PMB at the Ring and Ball softening point.

3.3. Bitumen properties at the softening point

As the iso-modulus approach does not lead to a common quantitative relationship with the softening point of both unmodified bitumen and PMB, other approaches must be considered. For this, the $G^*$ and $\delta$ values of the studied binders at their respective Ring and Ball softening point temperatures were obtained by interpolating between the DSR results at 10 rad/s. The results are presented in Figure 6, where Figure 6(c) shows the results in the Black space. It is confirmed by these results that, at the Ring and Ball softening point, the original SBS-modified bitumen tends to show a lower $G^*$ and a lower $\delta$ than the unmodified bitumen, although the one with 3% SBS was closer to the unmodified binders. The original unmodified bitumen shows typically the $G^*$ at about 15 kPa and $\delta$ at about 82° at 10 rad/s. Previous studies (Alisov et al., 2020; Fan et al., 2014) reported approximately the same levels.

The RTFOT ageing increased the Ring and Ball softening point of all binders but did not change the $G^*$ at softening point very much (still about 15 kPa for the unmodified binders at 10 rad/s). However, lower $\delta$ values were observed after RTFOT for four of the five binders. The $\delta$ values of unmodified binders at the softening point all reduced to about 80°. The modified binder with 5% SBS showed a higher $\delta$ value at softening point after RTFOT ageing, possibly due to the damage of its polymer-rich network.

Based on these obtained $G^*$ and $\delta$ results, the values of three candidate criterion parameters were calculated at 10 rad/s for identifying a quantitative relationship with the softening point, including the elastic shear modulus $G'$, the previously reported $G^*/\sin\delta$ and the parameter usually used for the Glover-Rowe parameter $G'/\tan\delta$. The calculated results are plotted against the Ring and Ball softening point in Figure 7. It is indicated that the modified binder with 5% SBS still shows a much lower $G'$ than the other binders, although the difference between them seems reduced as compared to the difference in $G^*$. As expected, the $G^*/\sin\delta$ results did not make a very big difference from the $G^*$ results as shown in Figure 6(a). Nevertheless, the parameter $G'/\tan\delta$ shows the minimum difference between the unmodified bitumen and modified bitumen. The modified binder with 5% SBS is almost at the same level as the unmodified binders. This is because the PMB tends to compensate the ratio $G'/\tan\delta$ with simultaneously lower $G^*$ and lower $\delta$. The factor $\tan\delta$ is highly sensitive to the phase angles around
Figure 6. Complex shear modulus $G^*$ and phase angle $\delta$ of bitumen at 10 rad/s at the Ring and Ball softening point: (a) complex shear modulus $G^*$; (b) phase angle $\delta$; and (c) in the Black space.

Figure 7. Values of candidate rheological parameters at 10 rad/s at the Ring and Ball softening point: (a) $G'$; (b) $G^*/\sin\delta$; and (c) $G'/\tan\delta$.

the softening point (greater than 60°). Thus, the parameter $G'/\tan\delta$ is able to capture the common characteristic of unmodified and modified binders and does show great potential to be quantitatively related to the softening point of bitumen.
3.4. Quantitative relationship with the softening point based on both $G^*$ and $\delta$

Towards a common quantitative relationship for both unmodified and modified binders, both the complex shear modulus $G^*$ and phase angle $\delta$ should be considered. The analysis presented in the previous section suggests that the rheological parameter $G'/\tan\delta$ seems promising as the criterion to identify a quantitative relationship. The possibility of quantitatively relating it to the softening point of bitumen is thus further investigated in this section. As the elastic shear modulus $G'$ also shows certain potential compared to the iso-modulus approach, the analysis of quantitatively relating $G'$ to the softening point is described in the Appendix to this paper, for information purposes.

According to Figure 7(c), the values of $G'/\tan\delta$ showed the minimum difference between the unmodified and modified binders. In order to specify a quantitative criterion level for the relationship with softening point, the $G^*$ and $\delta$ values of unmodified binders are taken as the equivalent inputs to determine the $G'/\tan\delta$ level, because they are more stable and do not vary very much compared to the values of PMB. Since Figure 6(a) has indicated that the unmodified bitumen typically has the $G^*$ at about 15 kPa at the softening point, and the BTSV method has demonstrated its effectiveness at 10 rad/s for unmodified bitumen as well, the modulus level of 15 kPa is adopted in this study as the equivalent $G^*$ input to determine the quantitative criterion level.

Furthermore, as both Figures 5 and 6(b) have shown that the RTFOT ageing leads to reduced $\delta$ values for unmodified binders at the softening point (about 82° for original binders at 10 rad/s and 80° after RTFOT), it might be necessary to use different $\delta$ values to determine the criterion level before and after RTFOT. This can result in the most reliable quantitative relationship with the softening point. From the practical point of view, however, it is simpler and easier to use a common and uniform criterion level both before and after RTFOT if the uncertainty is acceptable. This would be beneficial to the estimation of softening point for unknown binders. Thus, a compromised average $\delta$ value (i.e. 81° both before and after RTFOT) can be of interest as well. Considering this, Table 2 lists the two quantitative criteria at 10 rad/s for the relationship with softening point of bitumen that are investigated and compared in this study, respectively denoted as Criterion 1 and Criterion 2. Criterion 1 takes the effect of RTFOT ageing into account for higher reliability while Criterion 2 is a compromised uniform criterion for both original and RTFOT-aged binders for practical purposes.

Based on the obtained DSR temperature sweep results, the parameter $G'/\tan\delta$ was calculated at 10 rad/s for all the studied binders and the $G'/\tan\delta$ curves were plotted, as shown in Figures 8 and 9 respectively for the original and RTFOT-aged binders. Logarithmic interpolation was applied for $G'/\tan\delta$ to determine the equivalent softening temperature $T_s_{G'/\tan\delta}$ at which $G'/\tan\delta$ is equal to the criterion values at 10 rad/s (linear interpolation for temperature). With Criterion 1, different $G'/\tan\delta$ values were used as the criterion before and after RTFOT, i.e. 293 Pa for original binders and 459 Pa after RTFOT. The determination with Criterion 1 is presented in Figures 8 and 9, and the obtained results are plotted against the Ring and Ball softening point in Figure 10. It is indicated that the equivalent softening temperature $T_s_{G'/\tan\delta}$ determined with Criterion 1 is a very good estimation of the Ring and Ball softening point.

### Table 2. Quantitative criteria at 10 rad/s for relationship with the softening point of bitumen.

<table>
<thead>
<tr>
<th>Criterion</th>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Original binder</td>
<td>Complex shear modulus $G^*$ (equivalent input)</td>
<td>15 kPa</td>
</tr>
<tr>
<td>After RTFOT</td>
<td>Phase angle $\delta$ (equivalent input)</td>
<td>82°</td>
</tr>
<tr>
<td>After RTFOT</td>
<td>$G'/\tan\delta$</td>
<td>293 Pa</td>
</tr>
<tr>
<td>Original binder and after RTFOT</td>
<td>Complex shear modulus $G^*$ (equivalent input)</td>
<td>15 kPa</td>
</tr>
<tr>
<td>After RTFOT</td>
<td>Phase angle $\delta$ (equivalent input)</td>
<td>81°</td>
</tr>
<tr>
<td>After RTFOT</td>
<td>$G'/\tan\delta$</td>
<td>459 Pa</td>
</tr>
<tr>
<td>Original binder and after RTFOT</td>
<td>Complex shear modulus $G^*$ (equivalent input)</td>
<td>15 kPa</td>
</tr>
<tr>
<td>After RTFOT</td>
<td>Phase angle $\delta$ (equivalent input)</td>
<td>81°</td>
</tr>
<tr>
<td>After RTFOT</td>
<td>$G'/\tan\delta$</td>
<td>372 Pa</td>
</tr>
</tbody>
</table>
Ball softening point, even for the modified bitumen with 5% SBS. Comparing with the iso-modulus approach (Figure 4), the accuracy of the equivalent softening temperature $T_{s,G'/\tan\delta}$ as an estimation of the Ring and Ball softening point is largely improved for the modified binders while the accuracy for unmodified binders is still high.

With Criterion 2, a uniform $G'/\tan\delta$ value was used as the criterion both before and after RTFOT, i.e. 372 Pa. The determination was in the same way as with Criterion 1. The obtained results are also plotted against the Ring and Ball softening point, as shown in Figure 11. Comparing with the iso-modulus approach (Figure 4), the estimation accuracy with Criterion 2 is also largely improved for the modified binders while the accuracy for unmodified binders is also high. To compare Criterion 1
and Criterion 2, however, quantitative evaluation of accuracy is needed, as the difference between them seems not very big according to Figures 10 and 11. This will be discussed in the following section.

Additionally, the $G\ast$ and $\delta$ values at the equivalent softening temperature $T_{S,G'/\tan \delta}$ at 10 rad/s were obtained by interpolating between the DSR results, as shown in Table 3. The results reveal that the determination with quantitative criteria of $G'/\tan \delta$ does target much lower $G\ast$ and lower $\delta$ levels for PMB than for the unmodified bitumen. This agrees with the analysis of bitumen properties at the softening point (Figure 6) and thus indicates the effectiveness of the quantitative criteria for a common relationship with the softening point of both unmodified and modified binders. PMB tends to compensate the ratio $G'/\tan \delta$ with the simultaneously lower $G\ast$ and lower $\delta$ values. The high sensitivity of factor $\tan \delta$ to the phase angles greater than 60° enables the parameter to be quantitatively related to the softening point of both unmodified and modified binders.
Table 3. Complex shear modulus $G*$ and phase angle $\delta$ of bitumen at the equivalent softening temperature $T_{s_{\text{equivalent}}}$ (10 rad/s).

<table>
<thead>
<tr>
<th>Property</th>
<th>$G*$ (Pa)</th>
<th>$\delta$ (°)</th>
<th>$G*$ (Pa)</th>
<th>$\delta$ (°)</th>
<th>$G*$ (Pa)</th>
<th>$\delta$ (°)</th>
<th>$G*$ (Pa)</th>
<th>$\delta$ (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bitumen 50/70</td>
<td>14,868</td>
<td>81.9</td>
<td>13,772</td>
<td>79.5</td>
<td>17,167</td>
<td>81.5</td>
<td>12,093</td>
<td>79.9</td>
</tr>
<tr>
<td>Bitumen 70/100</td>
<td>15,326</td>
<td>82.0</td>
<td>13,948</td>
<td>79.6</td>
<td>17,635</td>
<td>81.7</td>
<td>12,244</td>
<td>80.1</td>
</tr>
<tr>
<td>Bitumen 160/220</td>
<td>17,477</td>
<td>82.5</td>
<td>15,874</td>
<td>80.2</td>
<td>20,191</td>
<td>82.2</td>
<td>13,971</td>
<td>80.6</td>
</tr>
<tr>
<td>PMB 3% SBS + 100/150</td>
<td>8,631</td>
<td>79.3</td>
<td>7,557</td>
<td>73.8</td>
<td>9,796</td>
<td>78.7</td>
<td>6,678</td>
<td>76.5</td>
</tr>
<tr>
<td>PMB 5% SBS + 100/150</td>
<td>1,935</td>
<td>67.9</td>
<td>3,013</td>
<td>67.8</td>
<td>2,338</td>
<td>67.5</td>
<td>2,638</td>
<td>68.6</td>
</tr>
</tbody>
</table>

3.5. Accuracy evaluation and comparison of different criteria

As mentioned previously, a quantitative evaluation of accuracy is needed to compare the different criteria. For this, the inaccuracy of each determination was calculated, which is defined as the absolute value of the difference between the equivalent softening temperature and measured softening point by the Ring and Ball method (Sironi et al., 2018; Tangmose et al., 2015), i.e.

$$\text{Inaccuracy} = |T_{s_{\text{equivalent}}} - T_{R&B\,\text{measured}}|$$  (1)

The calculation results for the determinations with Criterion 1 and Criterion 2, as well as the iso-modulus approach with $G*$ equal to 15 kPa at 10 rad/s, are presented in Table 4. It is indicated that all three quantitative criteria have low inaccuracy (less than 2°C) for the unmodified binders. The determinations with the criteria of $G'/\tan \delta$ show significantly lowered inaccuracy for PMB, especially for the modified binder with 5% SBS.

To make straightforward and explicit comparisons, all the unmodified binders (both original and RTFOT-aged) were grouped together to calculate the average inaccuracy, defined as the average of all inaccuracy values by

$$\text{Average inaccuracy} = \frac{\sum |T_{s_{\text{equivalent}}} - T_{R&B\,\text{measured}}|}{n}$$  (2)

The calculation was also done for each of the two modified binders to get the average inaccuracy of the original and RTFOT-aged binders. The results are shown in Figure 12, revealing that the inaccuracy for the modified binder with 5% SBS reduced to about 5.5°C with the quantitative criteria of $G'/\tan \delta$ from more than 20°C by the iso-modulus approach. Between Criterion 1 and Criterion 2, Criterion 1 has slightly lower inaccuracy. This is because it uses more precise but a bit more complicated criterion values than Criterion 2. Overall, the difference in inaccuracy between them was no more than 1°C.

3.6. Reliability verification with more binders

Test results of the analysed binders above in this paper has demonstrated the effectiveness of the quantitative criteria of $G'/\tan \delta$ to be more accurately related to the softening point of both unmodified

Table 4. Inaccuracy of different quantitative criteria.

<table>
<thead>
<tr>
<th>Estimation approaches</th>
<th>Iso-modulus $G* = 15$ kPa (10 rad/s)</th>
<th>Determined with Criterion 1</th>
<th>Determined with Criterion 2</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Original (°C)</td>
<td>After RTFOT (°C)</td>
<td>Original (°C)</td>
</tr>
<tr>
<td>Bitumen 50/70</td>
<td>0.0</td>
<td>0.4</td>
<td>0.0</td>
</tr>
<tr>
<td>Bitumen 70/100</td>
<td>0.5</td>
<td>0.6</td>
<td>0.3</td>
</tr>
<tr>
<td>Bitumen 160/220</td>
<td>0.3</td>
<td>1.2</td>
<td>0.7</td>
</tr>
<tr>
<td>PMB 3% SBS + 100/150</td>
<td>5.2</td>
<td>2.1</td>
<td>1.2</td>
</tr>
<tr>
<td>PMB 5% SBS + 100/150</td>
<td>25.4</td>
<td>22.8</td>
<td>4.4</td>
</tr>
</tbody>
</table>
bitumen and PMB. To preliminarily verify the reliability of this relationship, test data of some more binders were obtained by searching in the literature (Edwards et al., 2010; Lin et al., 2019; Lu et al., 2014), including the Ring and Ball softening point and DSR temperature sweep data at 10 rad/s of 14 various binders both unmodified and polymer-modified, both original and after RTFOT. The same approach as described above in this paper was applied to these extra binders. The results determined with Criterion 1 are plotted against the Ring and Ball softening point in Figure 13, while results with Criterion 2 in Figure 14.

It is indicated by the results that the equivalent softening temperature $T_{s,G'/\tan\delta}$ is a good estimation of the Ring and Ball softening point for both the unmodified and polymer-modified binders. Not only for SBS-modified bitumen, but the estimations were also accurate for the modified binders with ethylene-vinyl acetate (EVA) copolymer and wax additive. Some higher inaccuracy could be seen for the SBS-modified binders with a very high Ring and Ball softening point (over 90°C). A possible explanation for this could be that their Ring and Ball softening point was tested in the glycerol bath, which introduces both a systematic difference from the measurement in the water bath and larger measurement uncertainty. Additionally, the difference in density between glycerol and water might result in different bitumen properties at the measured softening point in different baths. Thus, the investigated quantitative relationship in this paper, which was identified based on Ring and Ball softening point measurements in the water bath, has higher accuracy with the softening point lower than 80°C.
Care should be taken when the determination result is over 80°C, although one of the extra modified binders in Figures 13 and 14 was accurately estimated to be slightly over 80°C.

To compare Criterion 1 and Criterion 2, the average inaccuracy of each group of the binders in the left side of Figures 13 and 14 (softening point tested in water) was calculated by Equation (2). This enables a fair comparison. The average inaccuracy of all those binders was calculated as well. The results are shown in Figure 15. It is indicated that Criterion 1 leads to slightly lower inaccuracy than Criterion 2, although the determination with Criterion 2 would be simpler and easier to conduct in practice. The overall difference in inaccuracy between the criteria was no more than 1°C. This suggests that Criterion 2 would result in fairly good estimations in practice if there is no need for very high accuracy.

3.7. Implementation of the quantitative relationship in the Black space

Further verification of the identified quantitative relationship may be still needed with much more binders over a long period of time. But it has been possible already now to demonstrate its potential usefulness in linking the performance-related parameters of bitumen to the past experience based on
In Figure 16, the quantitative relationship (both Criterion 1 and Criterion 2) is implemented in the Black space and compared with other criteria, including the iso-modulus criterion $G^* = 15\,\text{kPa}$ at 10 rad/s and performance grading criteria (linear viscoelastic) for the high temperature. It should be noted that, in Figure 16, the parameter $G'/\tan\delta$ is denoted in the equivalent form $G^*(\cos\delta)^2/\sin\delta$. The plot indicates the approximate area (in grey) around $G^* = 15\,\text{kPa}$ where the Ring and Ball softening point of unmodified bitumen is usually located, as well as the approximate area (in pink) for PMB.

With the help of Figure 16, it can be seen that the Ring and Ball softening point of PMB may correspond to varying $G^*$ and $\delta$ levels. The softening point of slightly modified bitumen is probably located at the high-modulus (also high phase angle) end of the area, while the softening point of highly modified bitumen almost reaches the region of performance grading for the high temperature. It is worth noting that the $G^*/\sin\delta$ criteria are almost iso-modulus at the high-temperature end of performance grading, due to the usually high phase angle.
The past experience is that neither the softening point nor the performance grading parameter \( G^*/\sin \delta \) is a good indicator for the high-temperature performance of PMB, although they may work well for unmodified bitumen. Towards more effective technical parameters, a recent development in Europe takes the iso-modulus approach within the linear viscoelastic range (Alisov et al., 2020; Eckmann et al., 2016) and focuses on a few selected \( G^* \) levels at 10 rad/s, including 15 kPa and higher. Meanwhile, the American specifications (AASHTO M332 and ASTM D8239) have taken a step forward with the multiple stress creep and recovery (MSCR) test. To compare and validate these different methods towards new technical specifications and bitumen products, especially in European countries, extensive filed studies are needed. To these validation studies, this paper provides an access and link to the extensive past experience of these countries based on the empirical Ring and Ball softening point.

4. Conclusions and recommendations

Aiming to assist in easing the transition from the penetration-based bitumen specification to performance-related specification that is foreseeable in many countries, this paper investigates the quantitative relationship of DSR results with the softening point of both unmodified and modified bituminous binders. Both the complex shear modulus \( G^* \) and phase angle \( \delta \) of bitumen are considered for the relationship. By interpolating between the DSR results, the bitumen properties at the Ring and Ball softening point were investigated. Three candidate rheological parameters were analysed for their potential as the quantitative criterion, including the elastic shear modulus \( G' \), the previously reported \( G^*/\sin \delta \) and the parameter usually used for the Glover-Rowe parameter \( G'/\tan \delta \). The analysis suggested that the parameter \( G'/\tan \delta \) is able to capture the common characteristic of unmodified and modified binders and thus shows great potential to be quantitatively related to the softening point of bitumen.

In order to quantify the criterion level of \( G'/\tan \delta \), the \( G^* \) and \( \delta \) values of unmodified binders at the softening point were taken as the equivalent inputs, because they are more stable and do not vary very much compared to the values of PMB. Two quantitative criteria of \( G'/\tan \delta \) at 10 rad/s for the relationship with a softening point were proposed and investigated for their effectiveness. Criterion 1 takes the effect of RTFOT ageing into account for higher reliability while Criterion 2 is a compromised uniform criterion for both original and RTFOT-aged binders for practical purposes. Logarithmic interpolation was applied for \( G'/\tan \delta \) to determine the equivalent softening temperature \( T_{s,G'/\tan \delta} \) at which \( G'/\tan \delta \) is equal to the criterion values at 10 rad/s (linear interpolation for temperature).

It is indicated by the results that the equivalent softening temperature \( T_{s,G'/\tan \delta} \) is a good estimation of the Ring and Ball softening point for both the unmodified bitumen and PMB. While keeping low inaccuracy for the unmodified bitumen, the inaccuracy of \( T_{s,G'/\tan \delta} \) as an estimation of the Ring and Ball softening point of PMB reduced largely with the criteria of \( G'/\tan \delta \) compared to the iso-modulus approach. Between the two studied criteria of \( G'/\tan \delta \), Criterion 1 resulted in slightly lower inaccuracy than Criterion 2, although the determination with Criterion 2 would be simpler and easier to conduct in practice. The overall difference in inaccuracy between the two criteria was no more than 1°C. This suggests that Criterion 2 would result in fairly good estimations in practice if there is no need for very high accuracy.

Besides the analysed binders in this paper, more binder data were obtained by searching in the literature to preliminarily verify the reliability of the identified quantitative relationship. The verification indicated that the relationship works very well for both the unmodified binders and PMB with the softening point below 80°C. Some higher inaccuracy was observed for the PMB with the softening point much higher than 80°C. One reason for this can be that the quantitative relationship was identified in this paper based on Ring and Ball softening point measurements in the water bath, but there is a systematic difference between the measurements in water and glycerol (used for softening point over 80°C with a larger uncertainty) baths. Thus, care should be taken when the determination result is over 80°C.
Moreover, the identified quantitative relationship is only preliminarily verified so far with a limited number of binders. Further verification and validation with more binders are still needed. In addition, the studied PMB in this paper is limited to SBS-modified bitumen. One modified binder with EVA copolymer, and two with both SBS and wax were included in the preliminary verification and the determinations were accurate. It is an indication that the identified quantitative relationship would work for polymer-modified binders with plastomer modifiers and wax additives. However, for modified binders with other types of modifiers, e.g. crumb rubber, further investigation is still necessary.

Last but not least, this study is limited to original and RTFOT-aged binders based on petroleum bitumen. The accuracy and reliability of the identified quantitative relationship for other types of binders and more complex binders with new types of additives need to be further investigated before a related conclusion can be drawn. As ageing may affect the phase angle of bitumen at the softening point and the identified quantitative relationship in this paper does consider the phase angle, how to incorporate the influence of long-term ageing and field ageing into the relationship would be a very important research question to estimate the properties of binders containing recycled materials, e.g. reclaimed asphalt pavement (RAP).

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**References**


**Appendix**

**Quantitatively relating the elastic shear modulus $G'$ to the softening point of bitumen**

Based on the obtained DSR temperature sweep results, the elastic shear modulus $G'$ was calculated at 10 rad/s for all the studied binders and the $G'$ curves were plotted, as shown in Figures A1 and A2 respectively for the original and RTFOT-aged binders. Logarithmic interpolation was applied for $G'$ to determine the equivalent softening temperature $T_{\text{G'}}$ at which $G'$ is equal to the criterion values at 10 rad/s (linear interpolation for temperature). Based on the input $G_0$ and $\delta$ values for Criterion 1 in Table 2, different $G'$ values were used as the criterion before and after RTFOT, i.e. 2,088 Pa for original binders and 2,605 Pa after RTFOT. This leads to higher reliability than the compromised uniform criterion. The determination is presented in Figures A1 and A2, and the obtained results are plotted against the Ring and Ball softening point in Figure A3.
Figure A1. The $G'$ curves at 10 rad/s and determination of equivalent softening temperature $T_s, G'$ of original bitumen.

Figure A2. The $G'$ curves at 10 rad/s and determination of equivalent softening temperature $T_s, G'$ of RTFOT-aged bitumen.

It is indicated that the equivalent softening temperature $T_s, G'$ determined with $G'$ is a good estimation of the Ring and Ball softening point for the unmodified binders, both before and after the RTFOT ageing. Even for the modified bitumen with 3% SBS copolymer, the $T_s, G'$ was close to the measured softening point. However, for the modified bitumen with 5% SBS, the difference between $T_s, G'$ and the measured softening point was large, i.e. an about 15°C difference. Comparing with the iso-modulus approach (Figure 4), the accuracy of $T_s, G'$ as an estimation of the Ring and Ball softening point of modified binders is slightly improved. Comparing with the determinations with $G'/\tan \delta$ (Figure 10), however, using $G'$ leads to much higher estimation inaccuracy for PMB.
Figure A3. Equivalent softening temperature $T_{s,G}$ versus measured softening point by the Ring and Ball method.