ABSTRACT. It is an ongoing challenge in the flexible pavement industry to create new and improved modifications to construction materials. A concept with growing popularity in many parts of the world is the addition of recycled ground tire rubber (GTR) to the liquid asphalt component of asphalt concrete pavement. However, modification with GTR has not been accepted nationally and several highway agencies remain skeptical about performance of this material. This skepticism is sustained by the variability in current methods of use associated with GTR modification.

This study focused on the evaluation of various test methods that are used to characterize GTR modified asphalt binders’ viscoelastic properties and investigating the variability in measured properties with respect to the change of the measuring geometry type. Detailed binder testing indicates that current Superpave test methods are not adequate to capture the rheological behavior of GTR modified binders. Meanwhile, the cylindrical system was able to capture rheological properties of GTR modified binders successfully at wide range of temperatures.

KEYWORDS: GTR modified binder, rheological measurement, cylindrical geometry, DSR
1. Introduction and Problem Statement

In recent decades, due to increasing costs associated with conventional asphalt binders, a number of low-cost asphalt modification and extension techniques have been introduced to optimize cost and performance of conventional binders (Bahia et al., 2001). This is generally accomplished with the addition of polymer additives to enhance a component of the material, such as asphalt binder within asphalt concrete (Nicholls, 1998). From the engineering point of view, polymer modified asphalt (PMAs) are materials with superior rheological properties with respect to the unmodified asphalt binders, and their use in the construction of modern asphalt pavements is increasing especially for high traffic volume roadways (Read et al., 2003; Airey et al., 2004). Addition of these polymers has shown improvement to asphalt binder properties; however, there has been growing uncertainty about the cost and future supply of these materials. This uncertainty has caused a need to find alternate modification techniques. A concept with growing popularity in many parts of the world is the addition of recycled ground tire rubber (GTR) to the liquid asphalt component of asphalt concrete pavement (Bahia et al., 2013).

GTR is a vastly available resource produced from used vehicle tires that if left unused will fill many of our landfills. Studies to date have shown that the addition of GTR may result in similar modification to asphalt binders as those with polymers (Bahia et al., 1994; Oliver, 2000; Bahia et al., 2013). There are many reasons to use rubber modified binders in asphalt pavements. In general, these modified binders have better performance properties than those of the base binders.

However, highway agencies remain doubtful and believe that superior pavement performance incorporating GTR is only applicable in specific conditions. Also, using new GTR materials with larger particles and higher percentages have brought other concerns about suitable method to test these materials. These uncertainties are sustained by the variability in current methods of use associated with GTR modification. Willingness to invest in additional test and equipment needed for specific methods are limited by lack of confidence and justification of those methods.

Current asphalt binder characterization is based on the Standard Specification for Performance Graded Asphalt Binder (AASHTO T315; ASTM D6373). However, this specification may have limitation when it comes to particulate materials with large particle sizes (Houston et al., 2014). In order to accommodate for testing these materials, the standard gap size needs to be changed to larger values. Changing the gap size can have significant effect on the measurement and it would change the system from absolute to relative (these concepts will be explained in the next section). Therefore, the main goal of this study is to reveal the practical and rheological challenges concerning the measurement of rubber modified asphalt
binder with a dynamic shear rheometer and to provide rheological background information to support the established and ongoing research in the asphalt industry.

1.1. Understanding Absolute and Relative Measuring Systems

"Absolute units" means, that the calculation of the rheological parameters (e.g. shear viscosity, complex shear modulus) on the basis of the raw measurement data which are indeed measured by the instrument (torque, angle, time) are independent of the individual design (e.g. dimensions) of the used measuring system. Therefore, rheological parameters can be compared directly to each other, as they are independent of the used measuring system. This is only possible if standardized absolute measuring systems are used. Therefore, several basic conditions need to be satisfied, especially concerning the required laminar shear conditions. One general requirement is that the shear gap (in a parallel-plate system the distance between the lower and upper plate) needs to be "narrow". Details can be found in the standards ISO 3219, ISO 6721-10 and DIN 53019-1 which define various measuring systems that are used in combination with a dynamic shear rheometer (DSR).

Geometries that do not meet the required conditions according to the standards are so called "relative measuring systems". Rheological parameters can be calculated, but should not be compared to measurements performed with different measuring systems, as the shear conditions are not defined. Measurement results with relative systems should only be compared when measured under equal conditions and thus with an equal measuring system. Many measuring systems in use today (e.g. stirrers, spindle geometries) are relative measuring systems as the required narrow shear gap dimensions cannot be specified. Relative systems are still used because of historical reasons or to measure samples that cannot be measured accurately with absolute systems, e.g. due to big particles in the sample or due to wall slip effects. Sometimes, it is necessary to compromise in the design to be able to meet the requirements of "real world" samples.

The new concentric-cylinder systems used in this investigation are relative measuring systems, as the shear gap needs to be large in order to be able to measure samples with rubber particles up to 2 mm size. However, apart from the large gap, the design of the systems is still very close to an absolute measuring system, allowing the comparison with parallel-plate (PP) systems that are widely used to measure asphalt binder with a DSR.

Besides, PP systems cannot be seen as absolute measuring systems without restrictions due to the shear rate distribution in the gap, especially when other basic parameters are not considered. Details can be found in ISO 6721-10 and DIN 53019-1. This must specifically be taken into account when discussing the use of large gaps up to 4 mm with PP systems.
1.2. Parallel-Plate (Plate-Plate, PP) Measuring Systems and its Limits

Parallel-plate (PP) measuring systems (geometries, see Fig.1) are described and defined by the standards ISO 6721-10 and DIN 53019-1. PP systems are widely used for rheological measurements with dynamic shear rheometers (DSR), not only for measurements of asphalt binders.

![Figure 1. Parallel-plate measuring system](image)

The main advantage of PP systems is the flexibility. They can be adapted in order to meet the requirements of various measurement requests concerning the sample, temperature range and other measurement parameters, e.g. by changing the plate diameter or the shear gap. The main disadvantage of PP systems is the non-constant shear gradient (shear rate distribution) in the shear gap since the value of the shear rate and the shear deformation is increasing from zero in the center of the plate to maximum at the edge of the plate. However, this effect is negligible when oscillatory tests are performed in the linear viscoelastic (LVE) region. Cone-plate (CP) measuring systems do not have this disadvantage due to the cone angle α.

ISO 6721-10 defines PP geometries in relation to the determination of the dynamic rheological properties of polymer melts, but these definitions can be seen as general recommendations, especially concerning the classification and usage of PP as an absolute system. According to the standards, for the distance H (gap size) between the lower and upper plate compared to the plate radius R is stated H << R. In other words: The measurement gap should be small compared to the plate radius. It is recommended to use plate diameters (2R) between 20 and 50 mm and gap sizes (H) between 0.5 and 3 mm. The ratio 2R / H should be in the range of 10 to 50. To give an example, the most frequently standard PP system used is the PP25 system with an upper plate diameter of 25 mm and a standard gap size of 1 mm. The ratio 2R / H for this system is 25 mm / 1 mm = 25. When fulfilling these basic conditions and using the calculations given in ISO 6721-10 and DIN 53019-1, measurement results are comparable between standard cone-plate, concentric cylinder and parallel-plate systems. It should always be kept in mind that a PP system with 8 mm diameter in combination with a gap of 2 mm, that is currently used as standard for the measurement of Pressure Aging Vessel (PAV), AASHTO R18, aged asphalt...
binder with a ratio of $2R/H = 8 \text{ mm} / 2 \text{ mm} = 4$, lies already outside the recommended parameters.

The larger the gap, the larger is the risk of undesired effects, e.g. transient behavior, edge effects and failure, migration off the gap and inhomogeneous deformation. Although these effects are more critical in rotational tests compared to oscillatory tests performed within the linear viscoelastic (LVE) deformation range, the recommendations regarding gap setting state a maximum gap (H) of 3 mm for PP systems (e.g. in ASTM D4440). However, only narrow gaps (compared to the plate diameter) allow the correct use of the standard definitions of rheological parameters based on the Two-Plates-Model when shearing under laminar flow conditions. The larger the gap (respectively the smaller the ration $2R/H$), the less favorable are the conditions for this assumption and the more the PP systems must be seen as a relative measuring system.

Another important issue is the maximum particle size. For reliable rheological measurements with PP systems, the gap size should be at least 5 times, better 10 times, larger compared to the largest particles, agglomerates or other rigid components in the sample. AASHTO T315 limits the size of particulate material in the asphalt binder to a maximum of 250 µm for the longest dimension, meaning that the gap size should at least be 4 times larger when working with a 25 mm diameter plate and 1 mm gap size. It is easy to understand that the particles in a sample should not be larger than the used gap size, as the particles would be squeezed (rubber particles) or the desired gap could not be set (solid particles), but even if the particles still fit into the gap, bigger particles influence the shear conditions in the gap. Indeed, shear stress, shear rate and shear strain are calculated based on the measuring system dimensions, including gap size. Particles in the sample reduce the free gap size which is available for the matrix fluid to flow in laminar layers as requested in theory and therefore are leading to incorrect results. As long as the particles are small compared to the gap, the effect is negligible. If bigger particles are present in a sample, the calculated rheological parameters must be seen as a relative values. In other words: the measurements can be performed and rheological parameters can be calculated, but the results are influenced stronger as usual by the used measuring geometry.

The idea is to increase the PP gap size to measure samples with bigger particles seems to be a logical step (see Fig.2). No new measuring systems and accessories are needed and the PP system is approved to measure asphalt binder. However, the kind of theoretical issues mentioned above lead to visible and understandable problems concerning sample handling and measurement practice in the lab.
Another big issue is the trimming behavior of rubber modified asphalt binder with particles > 1 mm. AASHTO T315-12 requests the formation of a slight bulge in the test specimen by trimming of excess binder and the final closure gap in order to ensure the accuracy of the diameter of the test specimen. The diameter of the sample is assumed to be equal to the diameter of the plates otherwise the calculation of the rheological parameters is incorrect. This cannot be achieved as even after trimming the edge area does not show a defined shape as can be seen in Fig.3.

Figure 3. Trimming of a rubber modified binder

To summarize the questionable points concerning PP systems with large gaps up to 4 mm, especially for rubber modified asphalt binder samples with particle sizes up to 2 mm:

- PP systems with gap sizes > 3 mm are not defined in any standard
- Unknown effects of gap sizes > 3 mm on the calculated rheological parameters (standard geometry factors are no longer valid)
- Sample sagging and leaking effects, leading to undesired time effects and incorrect rheological values
- Great number of rubber particles are directly in touch with the plates surfaces, possibly leading to increased direct friction forces
- Reproducible, accurate trimming is very difficult
- Increased influence of edge effects, as the edge area is greatly increased (it has to be noted the most influence on the measurement result with a PP system comes from the edge area
- There is no representative sample amount. Sample inhomogeneity may have big influence (especially on the phase shift angle $\delta$)
- Repeatability and reproducibility is limited
Some measurable consequences are shown in the Results and Discussions part of this investigation. The resulting requirements for a new measuring system for rubber modified asphalt binders are the following:

- Large gap to measure asphalt binder samples with rubber particles up to 2 mm (10 mesh) or even bigger
- Design of the geometry should be as close to existing absolute measuring geometries used with a dynamic shear rheometer (DSR)
- No time effect due to lack of binder or sagging effects
- No trimming influence due to large particles and due to large shear gap dimensions
- Reduced undesired influence caused by edge effects
- Measurement of a representative sample amount should be possible
- Easy handling for users

The approach shown in this investigation to meet these requirements is to use special concentric-cylinder ("cup and bob") geometries.

1.3. Concentric-Cylinder (CC) Measuring Systems (“Cup and Bob”)

Concentric-cylinder (CC) measuring systems consist of an inner cylinder (bob) and an outer cylinder (cup) (see Fig.4). CC systems are defined by the standards ISO 3219 and DIN 53019-1.

![Figure 4. CC system](image)

The main advantages of CC systems in general are the following:

- Samples cannot flow off the shear gap
- Good temperature control of the sample
- Less influence of evaporating solvents
- Disposable cups can be used to simplify cleaning
- No edge effects
- No trimming is necessary

One important disadvantage is that a relatively large amount of sample is required, leading to longer thermal equilibrium times compared to PP and CP systems (see part 3.1).

As long as the shear gap is relatively narrow, CC systems are classified as absolute measuring systems. The idea of these narrow gap standard CC systems according to ISO 3219 is to achieve values of shear stress and shear rate as constant as possible in the entire shear gap. In other words, the \( v(r) \)-curve (\( v = \text{velocity}, r = \text{radius} \)) is showing an almost straight line which is desired (see Fig. 5a). This allows the use of the standard definitions of the rheological parameters based on the Two-Plates-Model with laminar flow conditions, despite the rounded areas of the cylinder walls. The area can be considered to be relatively even. The larger the gap the less favorable are the conditions for this assumption, resulting in CC systems which do not meet the requirements as stated in ISO 3219 and need to be defined as relative systems. The \( v(r) \)-curve is showing a curvature which is not desired (see Fig. 5b). This may lead to inaccurate measurement results when testing non-newtonian samples, as the values of shear stress and shear rate are not constant throughout the entire shear gap, possibly resulting in secondary flow effects, flow instabilities, transient behavior and inhomogeneous deformation behavior (similar to what happens in PP systems with large gaps). However, the systems can be calibrated and verified with a certified standard oil to correct some of the effects, but it stays a relative measuring system, as the calibration is only 100% valid for the sample it was calibrated with.

![Figure 5. Narrow (a) and large (b) gap CC system](image)

ISO 3219 only specifies the ratio of radii \( \delta \) of the radius of the outer (or external) cylinder (\( R_e \)) to the radius of the inner cylinder (\( R_i \)). Absolute dimensions are not
specified. Please note that the ratio \( \delta \) is not connected in any way to the phase shift angle \( \delta \). Following, the ratio will be named \( \delta_{cc} \) ("delta-cc", see Fig.6) in accordance with Mezger (2006) to avoid confusions.

![Figure 6. Definition of \( \delta_{cc} \)]

The elementary requirement regarding the so called standard CC geometries (absolute systems) according to ISO 3219 is a ratio \( \delta_{cc} \) of 1.0847. A frequently used standard CC system is the CC27 system with \( R_i = 13.33 \text{ mm} \) and \( R_e = 14.46 \text{ mm} \), resulting in the desired ratio \( \delta_{cc} \) of 1.0847 and a gap size of 1.13 mm.

In order to meet the requirements to measure samples with particle sizes up to at least 2 mm, the gap size, respectively the ratio \( \delta_{cc} \), needs to be increased. As the increase of the external radius \( R_e \) is limited by the rheometer, the first approach is the use of a modified standard cylinder CC17 (bob diameter = 16.66 mm, \( R_i = 8.33 \text{ mm} \)) in combination with a standard CC27 cup (cup diameter = 28.92 mm, \( R_e = 14.46 \text{ mm} \)) that is normally used with a standard cylinder CC27 (bob diameter 26.66 mm, \( R_i = 13.33 \text{ mm} \)). This results in a gap size of 6.13 mm, allowing the measurement of samples with particles up to a size of 2 mm. Although the gap size of a CC system should also be at least 5 times larger compared to the largest particles (as previously described for the PP systems), it is a more reasonable approach then the use of PP systems. The ratio \( \delta_{cc} \) for this so called CC17SP system results in:

\[
\delta_{cc} = \frac{R_e}{R_i} = \frac{14.46 \text{ mm}}{8.33 \text{ mm}} = 1.74
\]

The calculation of rheological parameters using the formulas for standard cylinder systems according to ISO 3219 should only be used if 1.061 \( \leq \delta_{cc} \leq 1.22 \). If \( \delta_{cc} \) is outside of the boundaries the calculation is done according to DIN 53019-1. For ISO 3219 compliant systems the so-called "representative" rheological parameters are defined as related to the middle of the shear gap. DIN 53019-1 also contains formulas that are developed for large-gap cylinder systems, calculating the rheological parameters related to the bob surface. These calculations are mathematically corrections for a non-linear \( v(r) \)-function, but can only be seen as an approximation that is not valid for all kinds off materials. Therefore, as already
mentioned before, relative CC systems can be adjusted using a certified calibration fluid to measure correct absolute rheological parameters for the used calibration fluid. The calibration is performed to determine the geometry factors used for the conversion of raw data to rheological parameters. For other samples that show similar rheological behavior, the calibrated CC system will give results that are very close to the absolute value. Therefore the fluid used for the calibration should be chosen in close relation to the viscoelastic behavior of the "real" sample that is about to be measured with the selected geometry.

2.1.1. CC17SP Setup and Calibration

To reduce the necessary sample amount and temperature equilibrium times, shorter disposable CC27 "asphalt" cups can used in combination with the CC17SP bob. The diameter of the CC27 "asphalt" cup is equal to the diameter of the standard CC27 cup. A marking tool can be used to mark the height of the sample filling to improve repeatability and reproducibility. To be able to use CC systems with a DSR, a suitable CC temperature chamber is necessary. Existing PP temperature accessories cannot be used.

It is obvious that the CC17SP system can no longer be seen as an absolute CC system as defined by ISO 3219. Nevertheless, the increase of the ratio $\delta_{cc}$ is a necessary and inevitable compromise and due to the performed calibration, the measurement results for asphalt binder are very close to absolute values. For the adjustment of the geometry factors the viscosity standard Cannon N2700000SP (lot number 09101g) at 76 °C was used to be as close to the viscosity range of neat asphalt binder at higher temperatures as possible. The calibration with a low viscosity calibration standard would not fit as good to the rheological behavior of asphalt binder. After the adjustment the measurement result was verified in rotation (shear rate $1 \text{ s}^{-1}$) and oscillation (12 % strain, 10 rad/s angular frequency).

The reference dynamic viscosity ($\eta$) value according to the certificate is 127800 mPa·s. The verification result was perfectly within a range of ±1 % after the adjustment both in rotation and oscillation as can be seen in Fig.7.
The advantages of the new CC system compared to PP systems concerning the measurement of rubber modified asphalt binder are the following:

- No trimming issues, no edge effects, no lack of binder, no sagging effects
- Defined calibration of geometry factors allows the comparison of measurement results
- Special internal design to minimize possible temperature gradients in horizontal direction that are caused by the large gap
- Reduced cleaning effort due to the use of disposable cups
- Gap size is larger in relation to the particles
- Easier adaption to future measurement challenges, e.g. concerning even bigger rubber particles in the asphalt binder
- Measurement of a representative sample is possible due to the high sample volume

The main resulting disadvantage is the prolonged thermal equilibrium time that leads to an increased measuring duration.

### 2.1.2. PAV aged Asphalt Binder and Radial Compliance – CC10SP

Instrument radial (torsional) compliance is a common property of all dynamic shear rheometers (DSR), respectively the measurement geometries and the instruments itself. Generally speaking, only part of the angular deflection applied and measured at the optical encoder of the rheometer reaches the sample as the rheometer is not “infinitely stiff” and is twisted itself due to the applied torque during a measurement. The remaining part results in a (reversible) distortion of the mechanical components of the rheometer (the instrument itself and the measuring geometry). Measurement errors due to radial compliance effects are small as long as
the instrument radial compliance $Y_R$ is small compared to the sample compliance $Y_S$. In other words: if the rheometer and the measuring geometry are stiff compared to the sample.

If the exact total radial compliance of the instrument is known (given in rad/Nm), the effect can be corrected, resulting in correct absolute measurement values, even if the sample is getting stiffer. One method is to measure with the assumption of an infinitely stiff rheometer and run a correction after the measurement. The rheometers used in the present investigation use a more sophisticated approach as they take the actual rheometer compliance value automatically into account already during the measurement. The main advantage of this method is that the rheometer not only controls onto the desired sample strain but it also delivers directly the correct rheological properties such as $G^*$ and $\delta$ which correspond to the sample. If radial compliance effects are not taken into account, the reported values for the rheological properties like $G^*$ and $\delta$ may not be correct. The influence of instrument radial compliance can be reduced by decreasing the value of the ratio $Y_R/Y_S$, respectively the ratio between the deflection angle due to radial compliance $\varphi_{Compliance}$ and the sample deflection angle $\varphi_{Sample}$:

$$\varphi_{Encoder} = \varphi_{Compliance} + \varphi_{Sample}$$

and

$$\frac{\varphi_{Compliance}}{\varphi_{Sample}} = \frac{M \cdot Y_R}{M \cdot Y_S} \quad \Rightarrow \quad \frac{\varphi_{Compliance}}{\varphi_{Sample}} = \frac{Y_R}{Y_S}$$

with $M$ being the applied torque that can be canceled in the formula.

There are three theoretical ways to decrease ("improve") the value of the ratio:

1. Increase sample radial compliance $Y_S$
2. Decrease total rheometer radial compliance $Y_R$
3. Decrease measuring system surface

The third way is used when changing the plate diameter from 25 mm to 8 mm when measuring PAV aged asphalt binder with a dynamic shear rheometer as part of the SuperPave performance grading.

Preliminary tests have shown that another specially designed CC system is necessary to meet the measurement requirements of PAV aged samples. Due to the very high stiffness ($G^* > 10^8$ Pa) of PAV aged asphalt binder at low temperatures, the effect of radial instrument compliance needs to be taken into account particularly. Existing standard CC geometries are not designed to measure stiff samples, but rather to measure low-viscosity fluids. The previously described CCSP17 measuring system is also not suitable to measure PAV aged samples down
to low temperatures, even if the shaft thickness is increased and although the exact radial compliance value is known for this system. The ratio $Y_R/Y_S$ gets too high and the measurement results are not reliable anymore. To improve the ratio $Y_R/Y_S$, a special CC system based on the existing standard cylinder CC10 (absolute system) was designed to meet the measurement requirements of PAV aged asphalt binder sample. The total length and bob dimensions are the same as the standard CC10 bob to stay as close to the absolute system as possible. The use of a CC10 instead of a CC17 reduces the surface area and the thicker shaft leads to an approximately ten times stiffer measuring system (ten times less radial compliance). The thicker shaft also improves the robustness of the system, resulting in less risk of unintended, irreversible bending that can easily happen when measuring very stiff samples. Due to the same special internal design as the cylinder CC17SP, thermal gradients in horizontal direction due to the large gap are minimized.

As the shaft diameter is only slightly smaller compared to the bob diameter, the immersion depth of the system in the sample emerges as a new influence factor affecting repeatability and reproducibility. However, this can be handled by a defined filling and positioning procedure. The same CC27 "asphalt" cups as described before for the CC17SP system can be used as well as the same sample amount. The advantages listed above for the CC17SP system also count for the CC10SP system.

The use of the modified cylinder CC10SP (bob diameter = 10.00 mm, $R_i = 5.00$ mm) in combination with a CC27 cup (cup diameter = 28.92 mm, $R_e = 14.46$ mm) results in a gap size of 9.5 mm, allowing the measurement of samples with particles up to a size of 2 mm or even bigger. The gap size of this CC system is nearly 5 times larger compared to particles with a size of 2 mm in the sample and the ratio $\delta_{cc}$ results in:

$$\delta_{cc} = \frac{R_e}{R_i} = \frac{14.46 \text{ mm}}{5.00 \text{ mm}} = 2.89$$

The adjustment of the CC10SP system was performed similar to the adjustment of the CC17SP system described before, using the viscosity standard Cannon N2700000 (lot number 11101c) at 25 °C. The standard was used to be as close to the viscosity range of PAV aged asphalt binder as possible. After the adjustment the measurement result was verified in rotation (shear rate 0.01 s$^{-1}$). The very low shear rate is necessary due to the very small Newtonian range of the standard. The range was checked previously to the calibration.

The reference dynamic viscosity ($\eta$) value according to the certificate at 25 °C is 5151000 mPa·s = 5151 Pa·s. The verification result was perfectly within a range of ±3 % after the adjustment as can be seen in Fig.8.
2. Experimental Setup

For all measurements, the dynamic shear rheometer, SmartPave 102 and accessories from Anton Paar (Austria) were used. For the measurements with CC systems, the previously described CC17SP and CC10SP systems in combination with the disposable CC27 "asphalt" cups and the air-cooled cylindrical temperature chamber C-PTD180/Air were used, including a passive hood to reduce temperature gradients in vertical and horizontal direction. For the measurements with PP systems, the well-known standard asphalt PP systems with 25 mm and 8 mm diameter in combination with the suitable lower inset plates and the peltier controlled dry heating system with active hood were used. If not mentioned otherwise, measurements with PP25 (25 mm diameter plate) were performed with a gap size of 1 mm and measurements with PP08 (8 mm diameter plate) were performed with a gap size of 2 mm. Temperature calibrations with a temperature probe in the middle of the gap were performed for the PP and CC systems previous to the measurements to ensure correct sample temperatures over the complete used temperature range. If not mentioned otherwise, all tests were started after a temperature equilibrium time of 10 minutes (PP systems) respectively 35 minutes (CC systems) at the starting measurement temperature.

2.1. Samples and Sample Preparation

For the investigation of instrument related drift effects, the calibration standard Cannon N2700000 (lot number 11101c) was used.

The following neat (unaged) asphalt binders were selected and used in the present investigation:
- Base Asphalt PG64-22 without rubber particles
- Base Asphalt PG64-22 with 10\% 40/80 mesh ground tire rubber (GTR) particles (equal to particles sizes \(\approx 0.2\) mm and \(0.4\) mm)
- Base Asphalt PG64-22 with 10\% 30 mesh ground tire rubber (GTR) particles (equal to particles sizes \(\approx 0.6\) mm)
- Base Asphalt PG64-22 with 10\% 10 mesh ground tire rubber (GTR) particles (equal to particles sizes \(\approx 2\) mm)
- ARB 18\% CRM (passing mesh 10)

The exact particle size distribution of the rubber particles was unknown. The ground tire rubber (GTR) particles are rather flat than spherical, so one side can be much smaller than the given particle size. For the PG64-22 samples it was expected that all particles are as large as the given mesh size. This could be validated when extracting the particles from the binder with solvent. The extracted particles in these samples were equal in size. Additionally, the rubber particles swell up in the asphalt binder, resulting in even larger particles in the sample than the given mesh or particle size.

The following PAV aged asphalt binders were selected and used in the present investigation:
- PAV aged PG64-16 (no rubber particles)
- ARB (Asphalt Rubber Binder) 18\% CRM (Crumb Rubber Modifier, passing 10 mesh) - PAV 2 cycles

The average particle size of the rubber particles in the neat and PAV aged ARB 18\% mesh (passing 10 mesh) asphalt binders was (qualitatively valued) much smaller compared to the rubber particles in the unaged PG64-22, 10\% 10 mesh sample. The exact particle size distribution was not known.

If not mentioned otherwise, sample preparation was performed according to AASHTO T315-12. Silicon molds were used to pour pellets for the measurement with PP systems. For the measurements with CC17SP and CC10SP systems, the heated binder was directly poured into the disposable CC27 "asphalt" cups up to the mark. A new sample was used for each measurement. The exact measurement presets are described in the following part of the present investigation.

3. Results and Discussion

3.1. Reaching Thermal Equilibrium

Due to the much higher sample volume in the CC17SP system compared to the PP25 or PP08 system and due to the low thermal conductivity of bitumen of 0.2 W/m\(\cdot\)K (water has a thermal conductivity of 0.58 W/m\(\cdot\)K, both at room temperature), it takes at least 30 minutes to gain thermal equilibrium in the CC17SP.
and the CC10SP system. Nevertheless, it is essential to wait for thermal equilibrium, otherwise the measurement results are incorrect and time dependent. Fig. 9 presents the time dependent curve progression of the complex shear modulus $G^*$ when performing a temperature step from 52 °C to 40 °C. The Base Asphalt PG64-22 without rubber particles was used as a sample under constant oscillatory shear conditions (1 % strain and 10 rad/s angular frequency). It can be seen that the cooling time of the cylindrical heating device is higher compared to the plate heating device and that the time to reach stable $G^*$-values is much higher for the CC system. This is a physical limitation that cannot be eluded.

Figure 9. Thermal equilibrium times (PP25 and CC17SP system)

3.2. Measurements with PP systems

3.2.1. Leak of Asphalt Binder

In order to quantitatively investigate the time effect on the measurement due to the expected leak of binder when using PP systems with 4 mm gap size, a time test was performed with a PP25 system. The neat Base Asphalt PG64-22 with 10 % 10 mesh GTR was measured at a constant temperature of 76 °C and a constant measurement preset in oscillation (12 % strain and 10 rad/s angular frequency). To be able to take pictures of the sample, the active peltier hood was not used for this test. The test result is given in Fig. 10 and Table 1.
Figure 10. Leak of binder over time

The complex shear modulus, G*, shows decreasing trend over time. For a better illustration, a linear scaling of the G*-axis was chosen. Generally, changing G*-values can be a result of pending thermal equilibrium, changing measurement presets (e.g. in an amplitude or frequency sweep), a physical or chemical change of the sample (e.g. due to a curing or dry out reaction) or loss of sample. As the first three reasons can be excluded in this measurement, the decreasing G*-value clearly indicates the loss sample over time. The pictures taken during the measurement (see Fig.10) confirm this assumption. Table 1 shows the percentage deviation of the G*-values referred to the start value, underlining the significance of the undesired time effect.

<table>
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<th>t = 0 min</th>
<th>t = 30 min</th>
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<td>1712</td>
<td>1640</td>
<td>1567</td>
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<tr>
<td>Deviation [%]</td>
<td>/</td>
<td>- 6.1</td>
<td>- 10.1</td>
<td>- 14.1</td>
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</table>

Table 1. Deviation due to leak of binder over time

It can be assumed that the time effect would be even more serious when measuring an asphalt binder without rubber particles, as due to the adhesion of the binder to the rubber particles only part of the complete binder (mainly in the edge area) can leak out of the gap.

When measuring unaged rubber modified asphalt binder, the main issues concerning repeatability, reproducibility and reliability are the difficulties with trimming, the homogeneity of the sample in combination with the question about
representative sample amount, unknown effects of the rubber particles in a relatively narrow gap and the leak and sagging of binder, leading to unreliable measurement results with a PP system. Another issue may be the calculation of absolute rheological parameters, as can be seen in the following part of the present investigation.

3.2.2. Comparison of Measurement Results with 2 mm and 4 mm Gap Size

As described in the introduction part of the present investigation, PP systems with large gaps must be assumed to be relative systems. To investigate the influence of 2 mm gap size compared to 4 mm gap size on the rheological parameters, three repetition measurements at each gap size were performed with the PP08 system and then compared to each other. The PAV aged PG64-16 without rubber particles was used as sample to avoid the influence of rubber particles and a temperature step from constant 46 °C to constant 16 °C was performed. At 46 °C a constant measurement preset in oscillation (1 % strain and 10 rad/s angular frequency) was chosen. During the cooling procedure with maximum cooling rate, the strain was logarithmically decreased from 1 % to 0.01 % and then held constant at 0.01 %.

For the repetition measurements at each gap size, the coefficient of variation (CV) for the G*-value was calculated to evaluate repeatability (single-operator precision). The coefficient of variation is defined as the ratio of the standard deviation σ to the mean μ and expressed as a percentage. Fig.11 shows the three repetition measurements with the PP08 system at 2 mm gap size.

![Figure 11. Repetition measurements - PP08, 2 mm gap size](image)

As three repetition measurements are not enough data for reliable statistical statements, these CV-values must only be seen as a spot check within the context of the present investigation. Nevertheless, the obtained CV-values of 4.90 % at 46 °C and 6.02 % at 16 °C are very close to the CV-value of 4.9 % given in
AASHTO T315-12 for the single-operator precision for PAV residue (referred to $G^*\cdot\sin\delta$).

Due to the increased viscosity of PAV aged asphalt binder compared to neat binder and due to the lower measurement temperatures, herein leak of binder is not an issue. However, sagging effects of the sample could be observed and need to be taken into account. Fig.12 shows the three repetition measurements with the PP08 system and 4 mm gap size.

![Figure 12. Repeatability measurements - PP08, 4 mm gap size](image_url)

As expected, the obtained CV-values of 6.41% at 46°C and 6.59% at 16°C are slightly higher compared to the measurements with 2 mm gap size but are still in an acceptable dimension. Without rubber particles, a sufficient reproducible filling and trimming procedure can still be performed, depending on the experience of the user.

As the obtained CV-values indicate a sufficient repeatability in terms of PP08 systems with 2 mm gap size and 4 mm gap size, a comparison of the mean values is meaningful. Table 2 shows the deviation of the mean $G^*$-values at 46°C and 16°C. Several possible effects must be taken into account when discussing these high deviations, e.g. an overfilling effect of the gap at 4 mm (user influence) and incorrect calculations of the rheological parameters due to undefined shear conditions in a large gap, as the formulas are based on narrow gap conditions (as described in the introduction part). In contrast to CC systems, there are no alternative formulas for large gap conditions described in the standards for PP systems. Further investigations would be necessary to investigate these influence factors.
<table>
<thead>
<tr>
<th></th>
<th>Mean G* at 46 °C [Pa]</th>
<th>Mean G* at 16 °C [Pa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PP08, 2 mm gap size</td>
<td>1.45·10^5</td>
<td>1.96·10^7</td>
</tr>
<tr>
<td>PP08, 4 mm gap size</td>
<td>1.95·10^5</td>
<td>2.47·10^7</td>
</tr>
<tr>
<td>Deviation [%]</td>
<td>+34.5</td>
<td>+26.0</td>
</tr>
</tbody>
</table>

Table 2. Deviation between PP08, 2 mm gap size and PP08, 4 mm gap size

Nevertheless, even if repeatability and reproducibility are within acceptable limits when measuring PAV aged samples with a PP08 system at 4 mm gap size, the results have to be questioned in terms of absolute rheological values. A possibly necessary calibration of the system (e.g. with the viscosity standard Cannon N27000000SP) could be problematic due to the leak or sagging of the calibration standard.

### 3.3. Comparison between PP25 and CC17SP Measurements

#### 3.3.1. Test Parameters

The positioning (immersion into the sample) of the CC17SP bob was performed at 100 °C and then the sample was cooled down to start temperature. The preheating of the binder in the cup could also be performed in an oven. To keep the sample preparation equal, the samples measured with PP25 were also heated to 100 °C previous to the measurements. For the comparison between PP25 and CC17SP, the unaged Base Asphalt PG64-22 with and without rubber particles was measured, using different gaps with the PP system.

Temperature steps (always 12 Kelvin) from constant 88 °C down to constant 40 °C were performed in the following measurements with PP25 and CC17SP. At 88 °C a constant measurement preset in oscillation (12 % strain and 10 rad/s angular frequency) was chosen. The strain was reduced at lower temperatures to reduce the influence of possible undesired edge effects with the PP system and to reduce the torque in the CC system (76 °C: 10 %, 64 °C: 5 %, 52 °C and 40 °C: 1 % strain). The angular frequency was always constant at 10 rad/s. As always only one measurement was performed, no statistically reliable statements are possible. However, the measurement results reveal plenty important and essential information to prove the applicability of the CC17SP system.

#### 3.3.2. Base Asphalt PG64-22, no rubber particles

To investigate the difference in absolute rheological values between PP25 at 1 mm gap size and CC17SP when measuring real asphalt binder samples without rubber particles, the Base Asphalt PG64-22 without GTR was measured first. In the
context of the existing SuperPave performance grading (PG) system this kind of binder is measured with a PP25 system at 1 mm gap size according to AASHTO T315-12. Fig.13 shows the comparison between the measurement results with the PP25 and CC17SP system. With due regard to the different thermal equilibrium times, the curve shapes are equal and the values of the rheological parameters $G^*$ and $\delta$ are very close, as the curves are overlapping.

![Figure 13. Comparison between PP25 and CC17SP (PG64-22, no GTR)](image)

As quantitative deviations are difficult to evaluate using the diagram, percentage deviations were calculated with the mean value of the last 10 measuring points at each temperature (PP25 values as reference). There is no significant deviation in the measured viscoelastic behavior ($\delta$-value, maximum +0.29 % at 40 °C), indicating that there are no serious temperature related differences between the systems. It has to be noted that the performed calibration of the CC17SP system has no influence on the $\delta$-value, as the actual shear conditions in the gap are not changed.

There are several influence factors to explain the slight deviation in the measured $G^*$-values (maximum +6.84 % at 88 °C), mainly sample preparation, filling and trimming errors and of course the different shear conditions in the gap. The calibration of the CC17SP may not be 100 % valid for asphalt binders over this wide temperature range (respectively viscosity range). Generally spoken, the repeatability of measurements with PP and CC systems need to be taken into account. There is always a slight difference between repetition measurements due to the influence of the user and possible inhomogeneity of the sample. Considering all these underlying influence factors and the wide temperature range, the accordance between the PP25 and CC17SP system is very satisfying.

3.3.3. Base Asphalt PG64-22, 10 % 40/80 mesh GTR and 10 % 30 mesh GTR

To investigate the influence of small rubber particles, the Base Asphalt PG64-22 first with 10 % 40/80 mesh GTR and then with 10 % 30 mesh GTR was measured.
Measurements with PP25 at 1 mm and 2 mm gap size did not show significant differences, therefore only the measurement at 1 mm gap size is shown. Fig.14 shows the measurements with 40/80 mesh GTR particles and Fig.15 shows the measurements with 30 mesh GTR particles.

![Figure 14. Comparison between PP25 and CC17SP (PG64-22, 10 % 40/80 mesh)](image1)

![Figure 15. Comparison between PP25 and CC17SP (PG64-22, 10 % 30 mesh)](image2)

The decreasing $G^*$-values and increasing $\delta$-values that can be observed with both systems at the beginning of the measurements at 88 °C can be interpreted as the initial alignment of the rubber particles in shear direction, as this effect was not observable in any measurement without rubber particles. A pre-shear step in oscillation within the linear-viscoelastic range may be suitable to avoid undesired influences on the actual measurement results.

The deviations concerning the $G^*$-values are comparable to the previous measurement of the asphalt binder without GTR. The maximum percentage deviation is -9.37 % at 40 °C (PG 64-22 with 10 % 40/80 mesh). Considering the
previously discussed influence factors, the accordance is still satisfying. However, the deviation of the δ-values at lower temperatures is noticeable for both asphalt binders. For both binders, the percentage deviation is constantly changing to higher positive values when decreasing temperature. For the binder with 40/80 mesh particles from +0.19 % at 88 °C to +1.98 % at 40 °C and for the binder with 30 mesh particles from -0.48 % at 88 °C to +1.73 %. In other words: the δ-values measured with the PP system are lower, indicating a more elastic behavior at lower temperatures compared to the CC17SP system. This trend was not observable when measuring asphalt binder without rubber particles. One possible interpretation is that as with decreasing temperature the stiffness of the binder increases, more shear energy is transferred to the rubber particles. Due to the smaller gap size of the PP system, this effect may be enhanced, leading to higher elastic effects, as the elastic behavior is yet dominated by the rubber particles at the selected measurement temperatures.

3.3.4. **Base Asphalt PG64-22, 10 % 10 mesh GTR**

To investigate the influence of rubber particles with particles sizes up to 2 mm, the Base Asphalt PG64-22 with 10 % 10 mesh GTR was measured. Measurements with PP25 at 1 mm, 2 mm and 4 mm gap size were performed. Fig.16 shows the G*-values and Fig.17 shows the δ-values of the measurements.

![Figure 16. Comparison between PP25 and CC17SP (PG64-22, 10 % 10 mesh) – G*](image-url)
It is obvious and no surprise that the measurement with PP25 at 1 mm gap size (red curves) differs heavily from the other measurements. The rubber particles in the asphalt binder do not fit into the gap and are squeezed when going to measuring position, resulting in a constant high normal force, higher $G^*$-values and lower $\delta$-values. The increase of gap size or the use of a different measuring system is inevitable for the measurement of asphalt binders containing such rubber particles.

However, the measurements at 2 mm (green curves) and 4 mm gap size (orange curves) also differ significantly from the measurement with CC17SP (blue curves), indicating higher $G^*$-values (stiffer sample) and lower $\delta$-values (high elasticity). The progression of the $\delta$-curve at 2 mm indicates that there are still squeeze or friction effects (e.g. because the particles are swollen in the binder, increasing particle size), as the $\delta$-value increases from 88 °C to 76 °C and the values are significantly lower compared to the measurement at 4 mm gap size. As expected, the progression of the $G^*$ and $\delta$-curves at 4 mm gap size is more realistic as the particles fit into the gap without being squeezed. Nevertheless, the values differ significantly from the values obtained with the CC17SP system. The less strongly increase of the $G^*$-curve and the lower $G^*$-value at 40 °C are most likely the result of the leak of binder over time.

The key question is which measurement result is more reliable (PP25 at 4 mm gap size or CC17SP). Considering all previously discussed and shown parts in the present investigation, the authors’ side with the result of the CC17SP system. However, further investigations are requested.
3.4. Comparison between PP08 and CC10SP Measurements

3.4.1. Test Parameters

The positioning (immersion into the sample) of the CC10SP bob was performed at 100°C and then the sample was cooled down to start temperature. The preheating of the binder in the cup could also be performed in an oven. To keep sample preparation equal, the samples measured with PP08 were also heated to 100 °C previous to the measurements. For the comparison between PP08 and CC10SP, the PAV aged PG64-16 without rubber particles was used to avoid the influence of rubber particles.

For all following measurements, a temperature step from constant 46 °C down to constant 16 °C was performed. At 46 °C a constant measurement preset in oscillation (1 % strain and 10 rad/s angular frequency) was chosen. During the cooling procedure with maximum cooling rate, the strain was logarithmically decreased from 1 % to 0.01 % and then held constant at 0.01 %. The strain was reduced to reduce the influence of possible undesired edge effects with the PP system and to reduce the torque in the CC system. Normal force was controlled to zero newton during all measurements.

Again, no statistically reliable statements are possible. However, the measurement results reveal plenty important and essential information to prove the applicability of the CC10SP system.

3.4.2. PAV aged PG64-16 (no rubber particles)

To investigate the difference in absolute rheological values and the effect of radial compliance between PP08 at 2 mm gap size and CC10SP when measuring PAV aged asphalt binder samples without rubber particles, the PAV aged PG64-16 without rubber particles was measured first. In the context of the existing SuperPave performance grading (PG) system this kind of binder is usually measured with a PP08 system at 2 mm gap size according to AASHTO T315-12. Fig.18 shows the comparison between the measurement results. Again, the different thermal equilibrium times between the PP and CC system are visible. With due regard to the different thermal equilibrium times, the curve shapes are equal and the values of the rheological parameters G* and δ are very close, as the curves are overlapping.
Again, there is no significant deviation in the measured viscoelastic behavior (δ-value, -0.67 % at 46 °C and +0.46 % at 16 °C), indicating that there are no serious temperature related differences between the systems (as seen before for the CC17SP system).

The same influence factors as previously described can be used to explain the deviations in the measured G*-values (+11.43 % at 46 °C and +2.12 % at 16 °C). The immersion depth of the CC10SP system has to be taken into account as additional influence factor. The investigation of the corresponding deflection angles during the measurement showed that the radial compliance of the CC10SP system has no negative effect on the measurement due to the applied correction. This is supported by the very low deviation at 16 °C. Considering all these underlying influence factors and the wide temperature range, the accordence between the PP08 at 2 mm gap size and CC10SP system is very satisfying.

3.4.3. Repeatability CC10SP – ARB 18 % CRM, PAV 2 Cycles

To evaluate the single-operator precision (repeatability) with the CC10SP system, three repetition measurements with ARB 18 % CRM, PAV 2 Cycles were performed and CV-values were calculated. The repeatability strongly depends on the sample preparation, as the cups need to be filled with sample up to the mark as exactly as possible. Otherwise the immersion depth of the CC10SP in the sample varies as the system is always positioned the same way in the sample automatically (to simplify the positioning procedure for the user), causing a change in the measured G*-value (higher values if the immersion depth increases). δ is not affected by the immersion depth, but strongly affected by temperature changes and inhomogeneity in the sample. Fig.19 shows the repetition measurements.
As three repetition measurements are not enough data for reliable statistical statements, the CV-values must only be seen as a spot check within the context of the present investigation. Nevertheless, the obtained CV-values of $+2.79\%$ at $46\,^\circ C$ and $+1.15\%$ at $16\,^\circ C$ indicate a very good repeatability. To obtain such low CV-values, the preparation procedure needs to be performed accurately.

4. Summary and Conclusion

The results presented in this investigation show that the calibrated concentric cylinder system (Bob & Cup) with large gaps is capable of measuring absolute rheological values compared to the widely used parallel plate systems for the measurement of asphalt binders. There is no absolute system available for asphalt binder samples with GTR particles up to a size of 2 mm or even larger. The one or the other compromise is inevitable in order to develop a reliable rheological method. However, when using specially designed measuring systems, the general disadvantages of relative systems can be minimized, enabling the development of a reliable and comparable test method for GTR modified asphalt binders.

The measurement of rubber modified asphalt binders with existing parallel plate systems at gap sizes larger than 2 mm leads to several theoretical and practical problems and questionable results. Filling, trimming, sagging, leak of binder and issues concerning the calculation of absolute rheological parameters need to be addressed and these factors would significantly affect the repeatability, reproducibility and reliability of the method.

The main question that has to be answered is the maximum rubber particle size that will be used as asphalt binder modifier. If only smaller rubber particles ($< 1\,\text{mm particle size}$) will be used, parallel plate systems may still be suitable to characterize
GTR modified asphalt binders’ viscoelastic properties, possibly using gap sizes up to 2 mm also with the PP25 system. Nevertheless, all issues addressed and investigated in this study need to be noted and taken into account when using and adapting the current Superpave test methods.

However, cylindrical geometry (Cup & Bob) is a reliable method to measure different GTR modified binder samples even with very large rubber particles. When using CC systems, the use of lower strains should be considered to prevent unnecessary high torques, especially when measuring PAV aged asphalt binder. As all measurements are expected to be in the LVE range of the asphalt binders, the measurement result is not affected when using lower strains. Further investigation concerning the LVE range of rubber modified asphalt binders should be conducted.

It is suggested that optimization steps (e.g. concerning sample preparation) as well as repeatability and reproducibility of the test method with the special concentric cylinder systems should be performed by the users as part of round robin tests. However, the development of a performance grading system for rubber modified asphalt binders based on concentric cylinder systems is feasible and within reach.

5. Bibliography


ISO 3219, “Plastics - Polymers/resins in the liquid state or as emulsions or dispersions - Determination of viscosity using a rotational viscometer with defined shear rate”, Standard by International Organization for Standardization, 1993.


