THE STRESS RELAXATION OF MODIFIED BITUMENS

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Abstract

The Asphalt pavements in Central and Eastern Europe have to be resistant to high and very low temperatures and climatic changes. Especially emphasize influence of low temperatures seems to be critical parameter. That is why the modified bitumens with low temperature susceptibility and with high resistance to low temperature cracking are searched for. Dynamic Shear Rheometer (DSR) can be used as a quick method that can evaluate the modification system of bituminous binders. The advantage of DSR testing is also related to possibility of comparison of unaged and aged bitumen.

The relaxations of shear stress of several asphalt rubber binders (containing 11 %, 13 %, 15 % and 17 % of crumb rubber) and paving bitumen were determined in DSR at the temperature of 0 °C and -10 °C. Relaxation tests were performed in the controlled strain regime. Total shear strain (rotation of upper geometry) was set to 1 % of the sample thickness and stress was applied for 60 s. Relaxation time was set to a period of 15 minutes. The shear stress relaxation behaviour of unaged bitumens and bitumens aged after 75 minutes and 225 minutes in Rolling Thin Film Oven Test (RTFOT) is presented and discussed.

Introduction

Bituminous binders in Central and Eastern Europe must resist a wide range of temperatures. In the Czech Republic, the temperature of the pavement surface when lit by the Sun can be as high as 60 °C, while during the winter months can fall even below -25 °C. High temperatures impose a risk of development of permanent deformations, whereas low temperatures can lead to crack formation. In the recent years, apart from binders modified with polymers, also Crumb Rubber modified Bitumen (CRmB or named as asphalt rubbers) are being used, which are quite common in for example the United States, the Republic of South Africa and to a limited extent also in some other European countries (Carlson and Zhu, 1999), (Heitzman, 1992), (Kudrna and Dašek, 2012).

The Issue of Low Temperature Properties of Bituminous Mixtures and Bituminous Binders

To evaluate the low temperature properties of bituminous mixtures, the Thermal Stress Restrained Specimen Test (TSRST) according to the EN 12697-46 (2012) is currently being used. During this test, a cuboid-shaped specimen, which length is held constant, is subjected to a controlled temperature decrease with constant temperature rate. Due to the prohibited thermal shrinkage, cryogenic stress is built up in the specimen until failure.

Figure 1. Thermal stress restrained specimen test - the different course of the curves for a mixture with paving bitumen and with CRmB

The assessment of low temperature properties provides information about the critical failure temperature and tensile strength for particular bituminous mixture (Isacsson and Zeng, 1998), (Sebaaly et al., 2001). Figure 1 shows the record of a TSRST test for two different bituminous mixtures, which both use a different bituminous binder. The first mixture contains a paving bitumen, the other one Crumb Rubber modified Bitumen (CRmB). The graph can be used to find the temperature region, at which there is still a complete relaxation of the tensile stress in the tested specimen (tensile stress does not increase as the temperature decreases) and it is also possible to derive a temperature threshold, at which this relaxation is no longer observed (linear relationship between stress and decreasing temperature – elastic behaviour). At higher temperatures (above approximately 10 °C), the mixture is able to eliminate the entire stress by stress relaxation, and in the temperature range from about 10 °C to -10 °C there is a transition from the relaxation phase to the linear phase. During the linear phase the mixture is no longer able to relax, behaves elastically and tensile stress increases until it reaches a level, at which a tensile crack forms in the tested specimen.

The TSRST single-axis strain test is time-consuming and the results depend especially on the thickness of the bituminous film, the air void content of the bituminous mixture, aggregate gradation, the type of bituminous binder used etc.

Another method used to assess the suitability of a bituminous mixture is based on Gauer test (Gauer, 1996), which is described in detail in the Technical

recommendations of the Czech Ministry of Transport TP 151 (2010). The test is based on determining the relaxation characteristics of a particular bituminous mixture using the tensile bending strength test. The test is performed in a water bath at a temperature of ± 0 °C. An example of results from a relaxation test of a bituminous mixture is shown in Figure 2. The characteristics of the bituminous mixtures used are given in Table 1. This test is also time-consuming and unable to reliably differentiate between the low temperature characteristics of the individual bituminous mixtures.

Table 1. Various bituminous mixtures used in the relaxation test

	Mixture	Binder content	Air void content	Bituminous film thickness	Residual stress in t = 300 s
		[%]	[%]	[10 ³ mm]	[%]
1	SAL 5	10,0	3,6	7.74	37,7
2	SAL 5	10,0	6,7	10.40	39,4
3	SAL 5	12,0	4,1	12.76	35,7
4	SAL 5	10,0	2,8	5.71	35,3
5	SAL 5	10,0	-	-	28,9
6	SAL 8	9,5	2,0	10.32	29,9

Note: SAL means Stress Absorbing layer with granularity 5 (8) mm

Because of the reasons outlined above, we were looking for an alternative method, which would easily and also effectively describe and compare low temperature characteristics of various bituminous binders in a lab. We focused on measuring the relaxation of shear stress of bituminous binders in a Dynamic Shear Rheometer (DSR). These tests were preceded by the standard bituminous binder tests.

Figure 2. The decrease in strength during the relaxation test of bituminous mixtures

Applied Test Procedures

The following empirical tests were selected to evaluate the properties of the measured bituminous binders: needle penetration according to the EN 1426 (2007), softening point using the ring and ball method according to the EN 1427 (2007), elastic

recovery according to the EN 13398 (2010), and penetration and recovery (resilience) according to the EN 13880-3 (2003). At the end, rheological tests were performed in the dynamic shear rheometer (DSR).

Rotational (oscillatory) rheometers (referred to in road engineering as Dynamic Shear Rheometers – DSR) are complex devices designed to characterize rheological behaviour of a wide spectrum of materials. They are used to describe the dynamic viscosity, and also allow measuring other rheological properties in both, the steady shear mode and the oscillatory mode. The main working part of a rotational rheometer consists of two co-axial geometries – a moving one (the rotor) and a static one (the stator) – while the tested material is placed between them. The geometries are used in three basic configurations: cylinder-cylinder, plate-plate, cone-plate, each of them appropriate for particular consistencies of the tested materials and different types of measurements (McGennis et al., 1994). For unusual materials (such as non-homogenous substances or substances containing larger solid particles), it is possible to use special geometry types – the vane tools (Zalman, 2011).

The tests were performed on the Kinexus rotational rheometer manufactured by the British company Malvern.

Measurement of the Shear Stress Relaxation in DSR

The measurement of the shear stress relaxation using the DSR can be an indicator, which shows the ability of a material to eliminate the imposed stress and also reflects the quality of the modifying system. The ability of a bituminous mixture to quickly absorb the imposed stress is a significant factor, which affects the ability of the bituminous mixture to resist crack formation. The ability of the bituminous mixture to transfer shear stress depends on the mixture properties, as well as the thickness of the bituminous film.

To determine the rate of shear stress relaxation in DSR, a procedure was used, where first a sample of a bituminous binder is exposed to a shear strain of a given shear rate under a given temperature. The shear strain should be in the linear viscoelastic region. The applied shear strain (rotation of upper geometry) is maintained at a constant level for the course of the entire test. After the initial increase of shear stress, which corresponds to the applied shear strain in applied shear rate, the decrease in stress over time is monitored. The result of the test is a relaxation curve of shear stress, which is the dependency of shear stress decrease on time.

For ideally elastic materials no shear stress relaxation occurs after a shear strain (rotation of upper geometry) is applied and the shear stress remains constant. In the case of a substance with ideally viscous behaviour, upon applying the shear strain, the shear stress is eliminated almost immediately. When shear strain is applied on a viscoelastic material, there is a delayed relaxation of the shear stress – either partial, or after a certain period of time, complete. The relaxation curve has an exponential shape and the degree of shear stress relaxation depends on the viscous component of the material.

When a shear strain (rotation of upper geometry) is applied on a viscoelastic liquid, a complete relaxation of the shear stress is achieved (provided that the relaxation time

is sufficiently long). In the case of viscoelastic solids, the shear stress relaxation does not reach zero even after a very long time period. The relaxation curve asymptotically approaches a final value, which is referred to as the equilibrium stress. This means that at least parts of the molecules are chemically or physically linked and therefore unable to move freely. It is possible to determine the relaxation module at any moment during the experiment by finding the ratio of the instantaneous shear stress and the initially set shear strain (Mezger, 2011).

Shear stress relaxation in DSR was determined at 0 °C and -10 °C using parallel plates geometry with a diameter of 8 mm and gap size 2 mm (Sui et al., 2011). This temperature was chosen with respect to the possible measuring range of the DSR and was derived from the measurements using the TSRST, as shown in Figure 1. Relaxation tests were performed in the controlled strain mode. The target shear strain (rotation of upper geometry) was set to a value of 1 % of the sample thickness and the time for reaching target strain was selected 60 s. Relaxation time was set to 15 minutes.

Laboratory Procedures Modelling Binder Ageing

One of the most common causes of formation of low temperature cracks is also binder ageing. Accelerated tests are used, which model short-term binder ageing during bituminous mixture production, and long-term ageing occurring on the real road (Brown and Scholz, 2000), (Mturi and O'Connell, 2012).

One of the most commonly used methods, which simulate short-term ageing, is the test of thermal stability in a thin moving layer by Rolling Thin Film Oven Test (RTFOT) according to EN 12607-1 (2007), the test of thermal stability of thin film by Thin Film Oven Test (TFOT) and the test of short-term ageing by Rotating Flask Test (RFT) (Bahia et al., 1998).

To test the long-term binder ageing, the most commonly used test is a test of accelerated long-term ageing in a pressure ageing vessel (PAV) or the Rotating Cylinder Ageing Test (RCAT) (Bahia and Anderson, 1995). It is also possible to use a modified version of the RTFOT, where the test period is tripled in comparison to the standard RTFOT used for short-term ageing. Such RTFOT modification can better differentiate between various binders and its tendency towards thermo-oxidative ageing (Muller and Jenkins, 2011). This 3-times-RTFOT-aging method was also used in our work (Spiegl, 2009).

The effects of heat and air are determined from the change in mass of the bituminous binder in flasks or change in bituminous binder properties, which are determined before and after the ageing process. To determine the changes in properties of the binder it is possible to use the needle penetration test, softening point or for example dynamic viscosity according to EN 13302 (2010). Changes can be described using rheological properties determined by dynamic shear rheometer (DSR). Leakage of highly viscous binder (CRmB) from the flasks was avoided by inserting rolled thin metal plate into flasks not to reduce air flow from the nozzle.

Materials Used

In the Czech Republic, the asphalt rubber has been used more and more often in the recent years. It is generally assumed that this type of binder is suitable for stress absorbing layers (SAL). For that reason, CRmBs with different content of crumb rubber have been made, which thereafter undergo a simulation of short-term ageing by the RTFOT according to EN 12607-1 (2007) and long-term ageing by the modified 3xRTFOT. Using empirical tests and tests performed on the DSR, it is possible to evaluate the effects of the rubber on ageing and on the rate of binder relaxation.

CRmB was prepared by blending paving bitumen of gradation 50/70 with crumb rubber of grain size 0/1 mm (ambient process) in a laboratory mixer at a temperature of 175 °C for 60 minutes.

Test Results

In the first part, the results from the empirical tests of the bituminous binders are given and the effects of ageing determined by the RTFOT and its modified version 3xRTFOT are described. Next section then provides results from tests performed using the DSR.

Needle Penetration, Softening Point, Elastic Recovery and Resilience

Table 2 shows the results from needle penetration, softening point, elastic recovery and resilience measurements for individual binders.

Table 2. Basic characteristics before ageing, after RTFOT and 3xRTFOT, for the CRmBs and a paving binder

Rubber content [%]	Ageing	Needle penetration	Softening point	Elastic recovery	Resilience
		EN 1426 [0,1 mm]	EN 1427 [°C]	EN 13398 [%]	EN 13880-3 [%]
0	-	57	47,4	-	-
	RTFOT	37	52,9	-	-
	3xRTFOT	26	59,1	-	-
11	-	45	55,2	40,0	14,7
	RTFOT	35	60,7	65,0	18,3
	3xRTFOT	27	69,4	67,0	20,3
13	-	42	59,7	53,0	20,0
	RTFOT	33	66,2	69,0	22,7
	3xRTFOT	25	70,3	72,0	23,3
15	-	39	61,2	55,0	21,3
	RTFOT	30	67,6	72,0	25,0
	3xRTFOT	24	73,1	72,5	25,7
17	-	35	64,7	60,5	26,7
	RTFOT	29	69,4	72,5	27,0
	3xRTFOT	23	73,5	73,0	27,3

Figure 3 shows the change in needle penetration during different ageing levels and for different contents of crumb rubber. The paving bitumen 50/70 has the highest penetration and as the content of crumb rubber increases, the binder becomes stiffer. After short-term ageing using the RTFOT, the bituminous binder stiffens significantly and the differences between the individual binders with different contents of crumb rubber are smaller. The lowest values of needle penetration are observed on binders that undergo the triple RTFOT. It is also obvious, that the stiffness of binders with increasing amount of crumb rubber changes least, while the stiffness of the general paving bitumen changes significantly.

Figure 3. Change in needle penetration for CRmB and neat bitumen before and after ageing processes using RTFOT and 3xRTFOT

Figure 4 shows the change in softening point depending on the binder ageing and on the crumb rubber content. It can be seen that increase in the amount of crumb rubber increases the value of softening point, which leads to a higher resistance of bitumen mixture against permanent deformation. After binder ageing using RTFOT and 3xRTFOT procedure, the value of the softening point increases.

Figure 4. Change in softening point of CRmB and neat bitumen before and after ageing using RTFOT and 3xRTFOT

Figure 5 describes the change in elastic recovery. The highest elastic recovery was observed in the case of CRmB with the highest crumb rubber content. This is because rubber has a high elasticity and therefore its higher amount increases the elastic recovery. It is immediately obvious that elastic recovery increases as the crumb rubber content in the binder increases and also the effects of ageing are smaller.

Figure 5. Change in elastic recovery of CRmB after ageing using RTFOT and 3xRTFOT

The test of bituminous binder resilience describes the regeneration during 20 s at 25 °C after strain application. For CRmB this value is higher than for polymer modified bitumens. This result can be explained by the elastic properties of rubber. As shown in Figure 6, increased amount of crumb rubber again contributes to higher

values of resilience and a crumb rubber content higher than 15 % minimizes the effects of ageing.

Figure 6. Change in resilience of CRmB after ageing by RTFOT and 3xRTFOT

All the above described tests provide information about binder properties at temperatures of 25 °C and higher. It is documented that paving binder stiffens during ageing and becomes more brittle, which negatively affects the formation of low temperature cracks, and modification by using crumb rubber significantly reduces this stiffening. This reduction of stiffness of CRmB can be partially influenced by the digestion of the rubber particles in hot bitumen during long term ageing (3xRTFOT).

Shear Stress Relaxation

Figure 7 shows the record of shear stress over time as stress relaxation for paving bitumen with a gradation of 50/70 at a temperature of 0 °C. It is obvious that bitumen aged using the RTFOT or 3xRTFOT has a higher shear stress and higher stiffness during the shear strain application, which has a size of 1 % in all measurements.

Figure 8 shows the record of shear stress over time for paving bitumen and for CRmB containing 15 % of crumb rubber. The bitumens were tested at -10 °C and 0 °C. It can be seen that lowering the temperature to -10 °C leads to a much higher stress value. At 0 °C, the stress measured for the paving bitumen is approximately half the value observed in CRmB. However at -10 °C, the stress for both bitumens is the same. This can be explained by the ageing of bitumen and by lower temperature sensitivity of crumb rubber.

Figure 7. The record of shear stress relaxation test at 0 °C of neat paving bitumen before and after RTFOT and 3xRTFOT ageing

Figure 8. The record of shear stress relaxation test at -10 °C and 0 °C for neat bitumen and CRmB with 15 % crumb rubber content

Figure 9 shows residual stress dependence (complementary to stress relaxation) on relaxation time. Initial shear stress was standardized to a value of 100 %. It is obvious, that as the temperature decreases and crumb rubber content increases, the decrease of stress slows down, i.e. the rate of shear stress relaxation decreases. Bitumen ageing also decreases stress relaxation, but ageing of CRmB has almost no effect at -10 °C.

To make it more clear, a relaxation time of 15 s and 900 s was chosen and results are summarized in bar chart graphs that represent the decrease of stress by the value of

shear stress (see Figure 10 and Figure 11). Results emphasize the effects of ageing of paving bitumen on relaxation during both temperatures tested and how the effects of ageing decrease as the content of crumb rubber content increases, when the crumb rubber content in CRmB reaches 13 %, the properties are basically identical.

Figure 9. Decrease of stress for neat bitumen and CRmB 15 % depending on ageing and temperature

Figure 10. Decrease of stress at time t = 15 s and temperature of 0 °C, depending on the crumb rubber content and binder ageing

Figure 11. Decrease of stress at time $t = 900$ s and temperature of 0°C , depending on the crumb rubber content and binder ageing

Figure 12 shows the dependency of residual shear stress on temperature for the relaxation time of 15 s for paving bitumen and CRmB with 15 % crumb rubber content. It can be seen that there is a stronger dependency of stiffness on temperature in the case of paving bitumen, because the curve representing the reduction of relaxation with increasing temperature, rises faster than in the case of CRmB.

Figure 13 represents the slope of the line of stress reduction with increasing temperature for all tested binders. This slope of the line is given by the tangent of the angle between the curves described in Figure 12 and the horizontal axis. The smaller the magnitude of the slope, the more suitable is the binder for designing mixtures that resist crack formation, because the critical stress, which causes disruption of the tested specimen by a crack, is reached at a lower temperature.

Figure 12. Dependency of stress decrease at time $t = 15$ s on temperature

Figure 13. Size of the slope of the line of various binders during a temperature change from -10 °C to 0 °C

This confirms the results of the measurements of low temperature properties of bitumen binders obtained from the TSRST (see Figure 1). At temperatures lower than -10 °C, the paving bitumen will have worse low temperature properties than CRmB and a crack will form sooner (at a relatively higher temperature).

Some fluctuations in the results obtained using CRmBs with different crumb rubber content prove the effects of the heterogeneity of CRmB samples, especially the results from testing at lower temperatures are slightly variable, and it is necessary to make several measurements, where anomalies are eliminated and an average from at least 3 measurements is taken.

Conclusion

The reduction of crack formation on asphalt pavements during freezing is a serious issue in harsh climate conditions of countries in Central and Eastern Europe.

Various tests of bituminous mixtures are used to describe their resistance to cracks, for example the TSRST according to the EN 12697-46 and relaxation of bending stress at 0 °C. These, however, are time-consuming tests and also depend on the particular mixture characteristics (aggregate gradation, air void content, type of the binder, binder content etc.) and it is not possible to reliably determine the effects of bitumen binder type on crack formation.

The article focuses on binder tests at low temperatures, including the effects of binder ageing. Penetration, softening point, elastic recovery and resilience were determined on a set of bituminous binders consisting of paving bitumen and crumb rubber modified bitumens (CRmB), always for the original bitumen, after short-term ageing RTFOT (75 minutes) and long-term ageing RTFOT (225 minutes). None of these tests was able to determine the behaviour of bitumens at low temperatures, and only based on the results of the ageing tests it was possible to predict questionable behaviour of binder.

Measuring low temperature properties using dynamic shear rheometer (DSR) proved to be much more reliable. A test of shear stress relaxation at temperatures of 0 °C and -10 °C was chosen, using parallel measuring plates with a diameter of 8 mm and gap size 2 mm. These temperature points were selected with respect to the possible measuring range of DSR and were derived from the measurements of low temperature properties of bitumen mixtures using TSRST. Relaxation tests were performed in the controlled strain regime. Shear strain (rotation of upper geometry) was set to 1 % of the sample thickness and stress was applied for 60 s. Relaxation time was set to a period of 15 minutes.

Binder tests, especially after the accelerated ageing simulation, were able to well differentiate between the behaviour of paving bitumen and bitumens with various crumb rubber content. A well-known practical experience that CRmB with crumb rubber content of 15 % provides suitable binder properties, reduces ageing and exhibits improved low temperature behaviour, was proved.

CRmBs could be a good alternative to polymer modified bitumens. In the Czech Republic, CRmBs are often used for stress absorbing layers (SAL), to overlay broken Portland cement concrete pavements or to overlay cracked bituminous layers with a thickness of 25 mm to 30 mm.

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