Creep Flow Behavior of Asphalt Rubber Binder. The Zero-Shear Viscosity Analysis

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ABSTRACT. *The development of the European technical specification aims at characterizing the asphalt binder behavior by means of rheological measurements and performance-related properties determination. In this context an experimental investigation was carried out at the University of Parma in order to characterize asphalt rubber binders by the Zero-Shear Viscosity (ZSV) analysis. This specific test highlights the binder performance in the operating conditions typical of hot climate, where the damages due to the viscous flow (rutting) are significant.*

In this paper the determination of Zero-Shear Viscosity (ZSV) in creep mode of asphalt rubber binders is presented and the theoretical and experimental critical aspects are highlighted.

KEYWORDS: *Creep, Zero-Shear Viscosity, Performance-Related Properties, Rutting*

1. Introduction

At present also in Europe, as it already happened in the United States, one can record an increasing determination to develop new technical specifications, as far as asphalt binder are concerned. At this point in time the rheological approach seems to be unavoidable in order to develop a correct system of classification that may include both traditional and special binders [BiT 03]. In fact, rheometry applied to bituminous binder allows the validation of physical and mathematical models that describe the behaviour of this kind of materials by quantifying - with an increasing accuracy - their reaction to stress [CHR 92, BAH 95]. On the basis of this concept many European countries have established research programs to the definition and the development of new rheometric test method, able to predict how binders will behave in operating conditions [STA 04]. Zero-Shear Viscosity is the rheological parameter identified and proposed to evaluate the anti-rutting performance of binders. This paper presents the application of the Zero-Shear Viscosity concept to asphalt rubber binders. In this context the paper aims at analysing the behaviour of bitumen modified by powdered tyre rubber on the basis of the creep test, first method indicated by the European researches for determining the Zero-Shear Viscosity of asphalt binder. This test was developed for traditional and polymer-modified binder, but were not applied to asphalt rubber binders yet, but the wide use of these materials calls for rigorous normative rules, in order to be classified, known and also qualified as construction materials according to the European Community directives.

In order to considered Zero-Shear Viscosity as an intrinsic property of materials, it shall not depend on the test conditions. Therefore this research also evaluate the validity of this assumption with a view to providing useful results for the development of the method and assessing the validity of the creep test as a tool to rank the performance of asphalt rubber binder.

2. Test instruments and materials

2.1 *Test instruments*

The tests were carried out by means of a Dynamic Shear Rheometer (DSR). The model used has a Peltier conditioning cell and an air-operated suspension system that reduces the friction between the moving parts. The selected measurement system, that defines the test configuration, consists in two plate sensors of 25 mm diameter with a gap of 2.0 mm. The test temperature was set at 60°C with a max. admitted deviation of $\pm$0.01°C from the selected temperature during the whole experiment. Before each test the samples was thermally conditioned between the plates for 30 minutes.

Figure 1. *Dynamic Shear Rheometer*

2.2 *Test materials*

The binders used in the experiment consist of traditional bitumen B50/70 (PG 64-22) modified by powdered tyres recycled in the laboratory at 0%, 16% and 20% rubber content referred to the weight of the bitumen, and not subjected to artificial ageing. So the tests were carried out for three different binders.

3. Experimental investigations

3.1 *Static creep test and the Zero-Shear Viscosity (ZSV) concept*

Zero-Shear-Viscosity is the viscosity of a material measured under particular conditions that allow it to become an intrinsic property of the material itself.

The concept of Zero-Shear-Viscosity (ZSV) has been developed in order to express the partial contribution of binder to rutting-resistance of pavements, because it was found that the SHRP rutting parameter $G^*/\sin\delta$ underestimates the performance of binders with an high delayed elasticity [PHI 96, DES 00, VIS 04].

The available literature describes many test methods to determine the ZSV, all of them based on the application of the Dynamic Shear Rheometer under different experimental conditions (both oscillatory and creep mode) [CLY 04, BIN 04].

The method used for this investigation, which is selected for standardisation by CEN, consists in applying a static stress, characterized by low entity ($\tau = \tau_0 \rightarrow 0$) and long application time, in order to reach the steady-state flow conditions ($d\gamma/dt \rightarrow$ cost). In this conditions the Zero-Shear Viscosity can be extrapolate, according to the theoretical equation of Burger model, by means of the measure of creep compliance J(t):

$$J(t) = J_0 + J_m \cdot \left(1 - e^{-\frac{t}{\lambda}}\right) + \frac{t}{\eta_0} \qquad (1)$$

where J_0 is the instantaneous compliance, J_m is the viscoelastic compliance, λ is the retardation time and η_0 is the Zero-Shear Viscosity. When long loading time is reached only the viscous modulus $J_v = t/\eta_0$ is increasing. So, according to the theoretical assumptions of this method, the ZSV of a material is represented by the inverse of the average slope that curve J(t) is assuming during the last minutes (e.g. 15 minutes) of the test according to the following equation:

$$ZSV = \Delta t / \Delta J = 900 / (J_f - J_{15}) \quad [Pa \cdot s] \qquad (2)$$

where J_{15} represents the compliance expressed in Pa^{-1} , measured 15 minutes before the load is no longer applied, J_f is the compliance expressed in Pa^{-1} , measured at the end of the test and 900 is the time interval between the two measurements expressed in seconds.

Here below we will examine the suggested approach and will highlight the theoretical assumptions and some experimental evidence that could be observed during the tests on bitumen modified by recycled tyre rubber.

3.2 *Organization and purpose of the experimental investigation*

The viscosity of asphalt binder depends of shear rate when applied stress is far to the zero-shear conditions [GAR 92]. So, in order to considered Zero-Shear Viscosity as an intrinsic property of materials, it shall not depend on the test conditions. This research aims at evaluating the validity of this assumption with a view to providing useful results for the development of the method and assessing the validity of the creep test as a tool to rank the performance of asphalt rubber.

During the investigation the creep test was repeated several times by varying the parameters that mainly define it, i.e. time (t) and tension (τ).

The range of variation was determined by keeping in mind that the creep time has to be long enough to guarantee that a steady flow state is achieved and that the tension applied will respect the zero-shear condition.

In accordance with the above, the parameters were defined as per table 1.

Table 1 *Reference parameters for the execution of the creep test*

Type of binder	**Stress, τ** [Pa]	**Time, t** [hours]	**Temperature, T** [°C]
Traditional Binder	10÷100	1	60
Asphalt Rubber Binder	10÷100	4÷8	60

4. Results

4.1 *Assessment of the Zero-Shear Viscosity*

The tests were initially carried out by imposing mean values to the parameters (τ = 50 Pa, t = 1 hour for traditional binder and τ = 50, t = 4 hours for asphalt rubber binder). On the basis of the results of this first phase, the influence on the final result of the variation of such parameters was determined.

At this stage, measures were taken to check the variation, as a function of time, of the most important variables that describe the behaviour of materials when it is subjected to actions in steady state: strain $\gamma(t)$, shear rate $d\gamma/dt$, compliance modulus J(t) and viscosity $\eta(t)$.

Different results were obtained in relation to the type of bitumen. For what concerns binder with 16% rubber content (Figure 1) the parameters trend was very similar to what can be typically measured by traditional bitumen (as for bitumen with 0% CRM). In this case one can say that the effect of the rubber is mostly evident in terms of viscosity increase whereas there is only a lightly deviation in the flow behaviour.

As for traditional binders, also in this case steady flow conditions arise, as testified by the fact that the slope of function J(t) is showing a constant value and that the curves referred to the three independent measurements overlap fairly well.

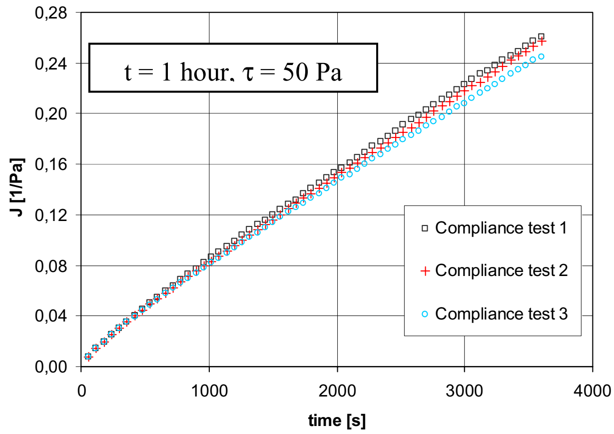

Figure 2. *Results of the creep test on binder with 16% CRM: evolution of compliance as a function of time (three independent measurements)*

Under these conditions, the viscosity measured in the considered lapse of time (i.e. the last 900 seconds of the test) tends to become constant: this value represents the ZSV of the material.

Like others properties of this kind of binders [ANT 04, LOH 00], the situation described above will change if the rubber content rises from 16% to 20%. By observing the evolution in time of the recorded variables, indeed, one can notice a change in the state of the flow (figure 3, 4) and, although the test has been continued for up to 4 hours, both viscosity and shear rate do not seem to tend to a value which remains constant in time (figure 3). The steady-state flow is not completely reached.

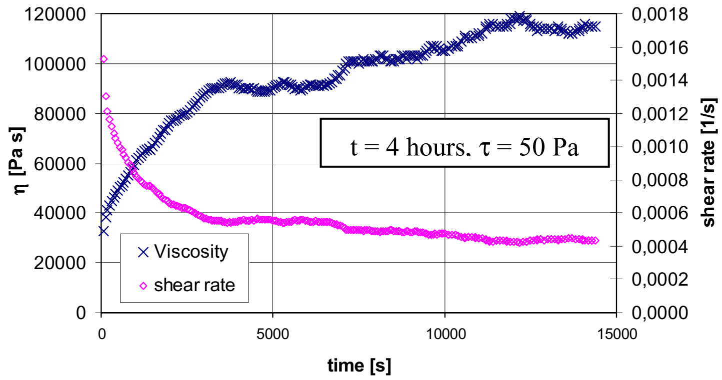

Figure 3. *Results of the creep test on binder with 20% CRM: evolution of viscosity and shear rate as a function of time*

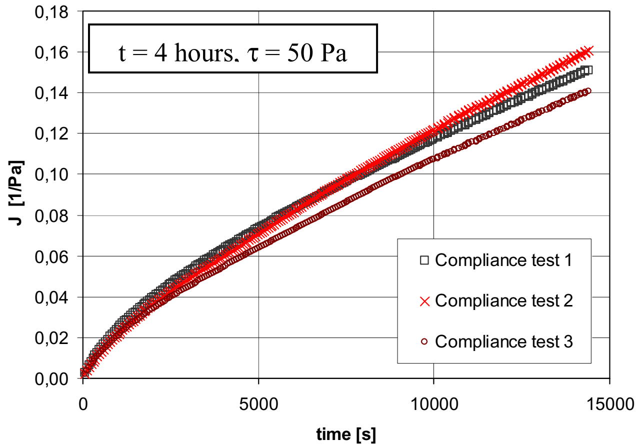

Figure 4. *Results of the creep test on binder with 20% CRM: evolution of compliance as a function of time (three independent measurements)*

The recorded trend, however, which can be ascribed to the very nature of the binder, did produce coherent and comparable results. In fact, the determination of the ZSV is based on the assumption that the first derivative of function J(t) calculated against time is a constant and that J(t) can be consequently approximated, within the calculated lapse of time, to a linear function such as

$$J(t) = at \qquad (3)$$

This is what actually happens also by bitumen with 20% CRM content, therefore the following equivalence can be considered valid:

$$\Delta t / \Delta J = 1/(dJ/dt) = \tau/(d\gamma/dt) = \eta \rightarrow ZSV \qquad (4)$$

In the case of bitumen with remarkably high tyre rubber content (20%) one can see that, during the last 900 seconds, the assumption of linearity for function J(t) can be considered correct, although the viscosity will not really come to a steady value [MON 06].

This is also demonstrated by the limited variation in the results of the three independent measures (table 2).

Table 2. *Results of the tests accomplished (three independent measures) in accordance with the considered method: T = 60°C, τ = 50 Pa*

Binder	***0% CRM***	***16% CRM***	***20% CRM***
ZSV test 1 [Pa s]	205	15517	134168
ZSV test 2 [Pa s]	214	15000	113982
ZSV test 3 [Pa s]	188	16364	132685
average [Pa s]	**202**	**15627**	**126945**
dev. [Pa s]	14	688	11251
dev. [%]	6,7	4,4	8,9

4.2 *Evaluation of the influence of the test parameters on the results*

In order to proceed with the investigation and ascertain the reliability of the results obtained by the creep test with binder at high CRM content (20% CRM), the tests were repeated by changing the essential parameters of time and stress.

4.2.1. *Influence of stress* - τ

The method proposed to standardize the static creep test, unlike in the case of traditional bitumen, does not propose, for modified binder, a unique value for the stress to be applied during the test. It provides for a range of values varying from 10 Pa to 100 Pa. This suggests how crucial it is to select the correct stress in order to get proper results.

What was found for asphalt rubber, indeed, is that by changing the stress, an increasing variability is recorded, both in the evolution of the values and in the final results, by increasing the percentage of modifying agent. (figure 5, 6).

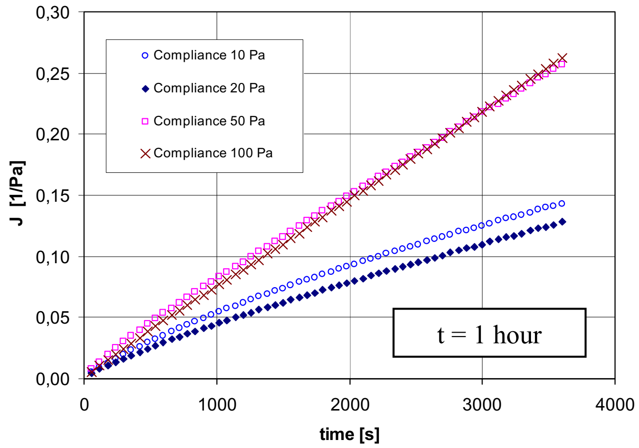

Figure 5. *Evolution of compliance at 60°C for bitumen with 16% CRM content at different stress values*

In the case of bitumen with 16% CRM content the level of stress will influence the test by causing a change in curve J(t) which can be recorded as a variation in both shape and final slope. At the lower levels of stress (10 Pa and 20 Pa) the curves will show a shape similar to that of highly modified bitumen.

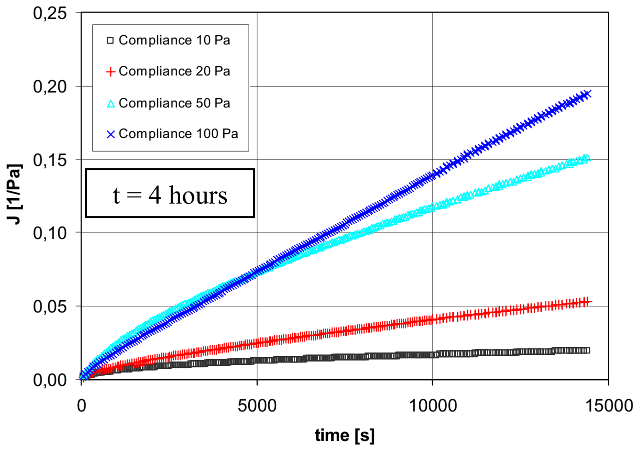

Figure 6. *Evolution of compliance at 60°C for bitumen with 20% CRM content at different stress values*

Also in the case of bitumen with 20% CRM content the slope of the asymptotic line is influenced by the entity of the stress (figure 6). Consequently, as shown in table 3, the influence of the stress will reflect upon the final test results.

Table 3 – *Measurements of ZSV according to the different stress levels applied (average of three independent measures)*

Binder	*Test Stress - τ*			
	10 Pa	**20 Pa**	**50 Pa**	**100 Pa**
CRM 0%	214	195	202	207
CRM 16%	32184	31667	15627	14066
CRM 20%	1809160	379010	126945	84583

For bitumen without CRM the variability of the test lies within the range of normal uncertainty which is typical for DSR tests [SAN 96]. On the contrary, in the further two cases (asphalt rubber binders) an increase in ZSV is recorded by reducing the stress. That becomes quite relevant when, as for bitumen with 20% CRM content, the test is accomplished at 10 Pa. In this case the recorded ZSV is passing from approx. $1.8 \cdot 10^6$ Pa s to approx. $1.3 \cdot 10^5$ Pa s, so becoming bigger by one order of magnitude compared to the value measured by $\tau = 50$ Pa. In the case of 20% CRM is possible to observe that there is not a linear relationship between shear stress and shear rate therefore, probably, a univocal value of ZSV doesn't exist.

4.2.2. *Influence of the creep time – t*

Another difficult issue when taking measurements on binder with high CRM percentage is how to determine the duration of the test. As seen in paragraph 4.2.1, even after 4 hours the viscosity of bitumen with high rubber content may still be on the increase. In such cases, therefore, ZSV could be underestimated.

In this phase of the investigation, several tests were executed on bitumen with 20% CRM with different creep times, i.e. 1, 2, 4 and 8 hours.

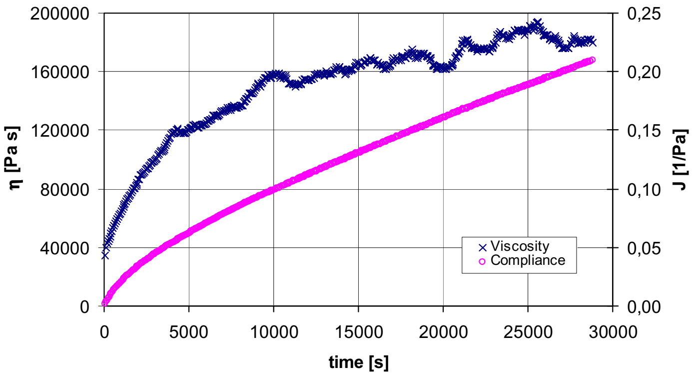

Figure 7. *Curves J(t) and η(t) obtained after 8 hours*

One can see (figure 7) that even after 8 hours (28800 s) no final result seems to be achievable since η(t) does not seem to tend to a constant value. Consequently, one can state that the slope of curve J(t), even after 8 hours, is constant only by first approximation.

In the case of bitumen with 20% CRM concentration, a steady-state flow (which is essential to get measures in line with the theoretical prescriptions) can only be achieved on condition that the stress is applied for a sufficiently long time: the duration of the test, therefore, appears to be, once again, a vital parameter also by CRM-modified bitumen.

The comparison shown in figure 8 defines the variability of the result based on the different creep times.

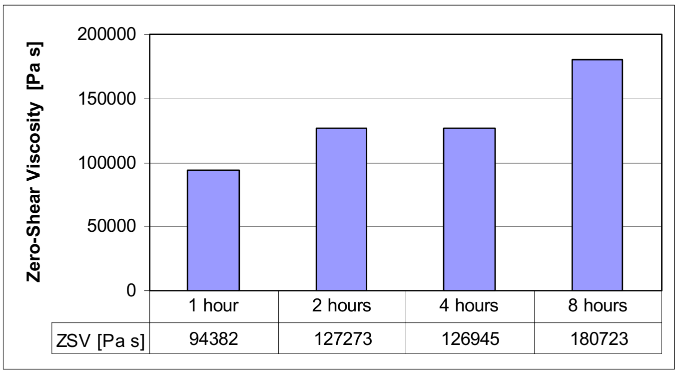

Figure 8. *Influence of the creep time on ZSV @ 60°C for bitumen with 20% CRM concentration*

5. Conclusions

The results of this article highlight several aspects that relate to the understanding of the mechanical behaviour of asphalt rubber binder, and also to the development of those methods which are typical of the rheological analysis applied to road construction. The definition of new technical specifications for asphalt binder at both European and US level is presently the centre of continuous and fruitful discussions and is therefore quite dynamic. So, the original contribution of this article consists in having pointed out, by experimental measures, some special and critical features which are essential for a correct application of the test on asphalt rubber binder.

Actually, through the analysis of the curves and the comparison of the final results, it has been pointed out that the selection of the test parameters may often lead to remarkable variations in the interpretation of the mechanical behaviour of material. Similarly to what is observed by PmB, the asphalt rubber binder with 20% CRM concentration will not reach steady flow conditions - at a low shear rate - in a rapid time. Whereas traditional binder is achieving a steady flow state in one hour, binder with high CRM content will need - nearly always - a longer time, not easy to define. Experimental investigation also shows that the definition of the applied shear stress is fundamental for the reliability of the result, because it has been demonstrate that the high rubber content causes a non-linear relationship between shear stress and shear rate near the zero-shear conditions too.

On the other hand, although it was conceived for traditional or polymer-modified bitumen only, the rheological approach can be applied with good results to bitumen modified by tyre rubber. In fact it has been demonstrated that the ZSV value is extremely sensitive to variations in the content of CRM within the binder, and that the flow conditions of the latter are varying according to the rubber percentage.

Furthermore, it has been underlined that the analysis of the curves is able to show the change caused by the addition of recycled tyre rubber on the behaviour of binder by shifting it towards the dominion of polymer-modified binders. In this sense one can see that the binder with 20% CRM concentration is showing a variation compared to the basic bitumen, and that the flow is changing from Newtonian to non-Newtonian, similarly to what is often recorded by binders modified by SBS polymers.

In any case, our investigation clearly shows that the ZSV numeric value is creating a distinction between traditional binders and binders modified by CRM rubber. This suggests that the Zero-Shear-Viscosity, despite the necessary approximation described above (particularly in the case of high rubber content), may represent a useful parameter to classify this kind of binders and make a forecast about their behaviour in some operating conditions.

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