New bitumen performance indicators - A feasibility study

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ABSTRACT

Since the 1999 Eurobitume Workshop, the search and validation of performance related bituminous binder properties continues to be a key issue for the paving industry in Europe, as well as in the rest of the world (especially for binders with complex rheological behavior). Important progress has been made and concepts such as the complex modulus measured with Dynamic Shear Rheometers (DSR) and low temperature stiffness and relaxation behavior measured with the Bending Beam Rheometer (BBR) have become familiar, along the lines of the Superpave system implementation in the US. Measurements in the linear domain of viscoelasticity have however also shown their limits and a new generation of binder tests, which try to directly address failure behavior, is now developing. Along with "conventional" rheology, three such test methods have been contemplated in this paper. In the low temperature domain, the ABCD (Asphalt Binder Cracking Device) test mimics the TSRST (Tensile Strength Restrained Specimen Test) performed on bituminous mixes. The LAS (Linear Amplitude Sweep) test is expected to be related to fatigue whereas the MSCRT (Multiple Stress Creep & Recovery Test) addresses high temperature performance. To evaluate the ability of each test method to differentiate between binders, the investigations have been based on three bitumen of the same penetration grade (70/100) but very different in chemical structure, varying from a "gel" to a "sol" structure (colloidal index of 0.24 to 0.09). Full rheology (DSR, BBR), MSCRT, ABCD and LAS tests have been conducted on all three bitumen, as well as on the corresponding RTFOT hardened and RTFOT+PAV aged binders. Further developments on the LAS test and its relation to fatigue performance are discussed in a separate paper by WRI.

Keywords: Ageing, Low-Temperature, Performance testing, Performance based standards, Rheology

1. INTRODUCTION

Since the 1999 Eurobitume Workshop, the search for and the validation of performance related bituminous binder properties has continued to be a key issue for the paving industry. Although conventional test methods do still have their value, a more fundamental approach based on rheological measurements is now advocated. Concepts like the complex modulus measured with Dynamic Shear Rheometers (DSR) and low temperature behaviour measured with the Bending Beam Rheometer (BBR) have become familiar. Measurements in the linear domain of viscoelasticity have however also shown their limits and a new generation of binder tests, which try to directly address failure behaviour in conditions expected to be closer to the field, occasionally out of the linear domain, is now developing. In the low temperature domain, the ABCD (Asphalt Binder Cracking Device) test mimics the TSRST (Tensile Strength Restrained Specimen Test) performed on bituminous mixes. The LAS (Linear Amplitude Sweep) test is expected to be related to fatigue whereas the MSCRT (Multiple Stress Creep & Recovery Test) addresses high temperature performance.

The objective of the study presented here was to evaluate and compare the ability of both conventional and more advanced rheological tests to differentiate bitumen known to be different in composition and structure. The investigation has been conducted on three bitumen of the same penetration grade (70/100) but of which the structure varied from a “GEL” to a “SOL” type. For each of them, testing has been done on the fresh bitumen, RTFOT and RTFOT+PAV aged binders.

2. INVESTIGATED BITUMEN

The bitumen used have been either sampled at the refinery or made in the laboratory while blending ad-hoc base products. They are therefore to be seen as representatives of “what might be observed” rather than as a panel of what is actually “on the market”.

As it can be seen in Table 1, these bitumen are markedly different in terms of composition (SARA analysis) with an n-heptane asphaltene content ranging from 12 to 7 mass-% and a colloidal stability index (Gaestel index) [1] ranging from 0,24 to 0,09. They vary thus from a “GEL” to a “SOL” structure. This has of course an influence on conventional physical properties. All three bitumen have the same penetration value at 25°C but differ significantly in terms of Softening Point and, hence, in the Pfeiffer Penetration Index. Large differences are also seen in Fraass brittleness temperature, to the advantage of the most structured bitumen.

Table 1: Characteristics of the investigated 70/100 bitumen

			SAMPLE		
			A	B	C
Characteristic	**Test method**	**Unit**	**GEL**	**SOL-GEL**	**SOL**
Asphaltenes	Thin Film Liquid Chromatography	% (m/m)	12,0	8,5	7,0
Resins		% (m/m)	16,0	15,5	13,0
Aromatics		% (m/m)	64,5	70,0	79,0
Saturates		% (m/m)	7,5	6,0	1,0
Colloïdal Stability Index	(Asph. + Sat.)/(Arom. + Resins)	-	0,24	0,17	0,09
Penetration at 25°C	EN 1426	mm/10	79	79	79
Ring&Ball Softening Point	EN 1427	°C	48,4	45,6	44,4
Pfeiffer Penetration Index	EN 12591 - Annex A	-	-0,47	-1,28	-1,65
Fraass brittleness temperature	EN 12593	°C	-24	-19	-13
Dynamic viscosity at 100°C	EN 13302	mPa.s	3696	2765	2639
Dynamic viscosity at 120°C		mPa.s	958	749	698
Dynamic viscosity at 140°C		mPa.s	335	274	245
Dynamic viscosity at 160°C		mPa.s	145	127	110

3. EXPERIMENTAL RESULTS – CONVENTIONAL PROPERTIES

As reported in Table 1, the conventional, so-called “empirical”, tests do of course reveal differences between the three binders and they are also sensitive to their evolution with ageing. This is much more pronounced for Softening Point and Fraass breaking point than for Penetration (Figure 1). Bitumen A (the most structured) has the highest Softening Point and lowest Fraass breaking point (largest plasticity index) but it also shows the largest changes with ageing, especially after RTFOT + PAV.

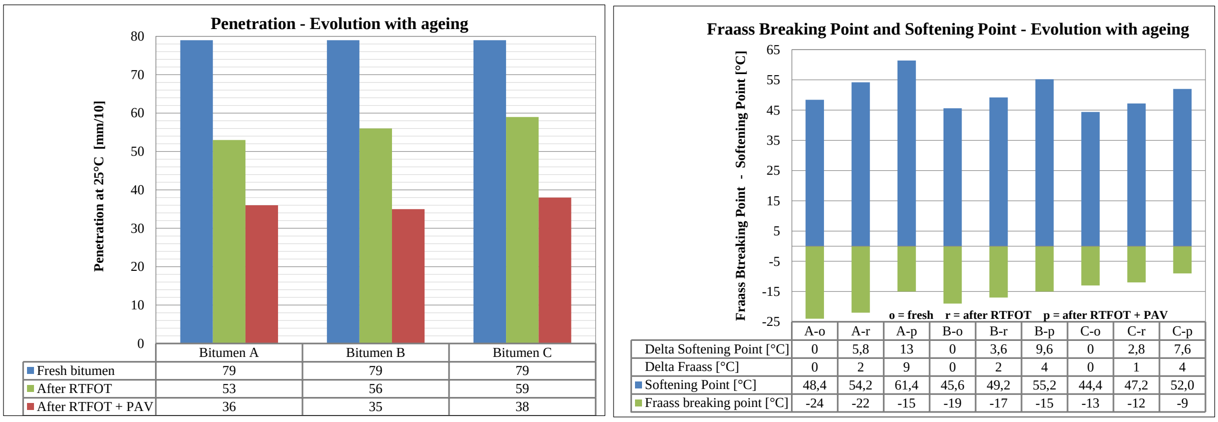
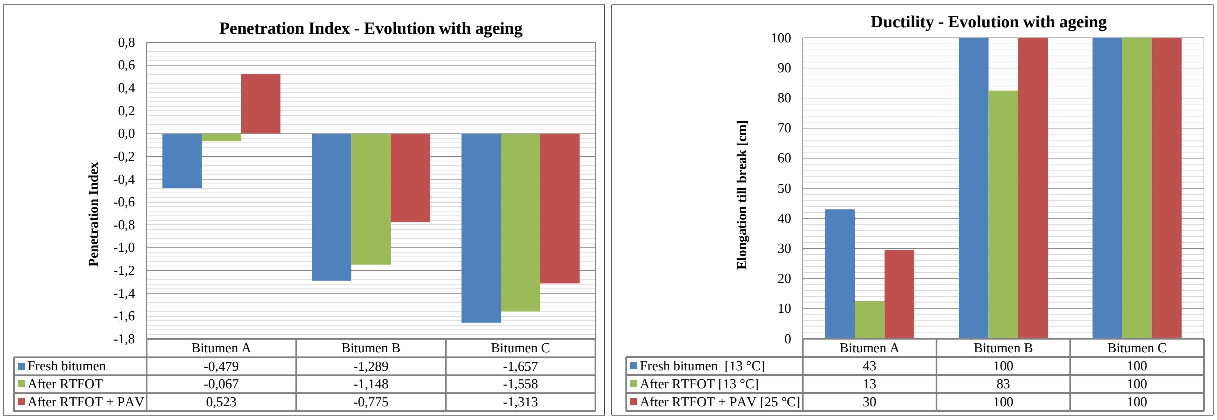


Figure 1a and 1b: Penetration, Softening Point and Fraass breaking point – Evolution with ageing

The Pfeiffer Penetration Index [EN 12591 – Annex A] also reflects quite strongly the structural differences between the 3 bitumen, as well as the strong evolution with ageing of bitumen A in comparison to B and C (Figure 2a). To guarantee a certain amount of deformation and the possibility to make comparisons, the ductility test [DIN 52013, ASTM D113] has been run at different temperatures (13°C for fresh and RTFOT aged samples, 25°C for RTFOT+PAV aged samples). The results (Figure 2b) clearly show lower elongation ability for bitumen A, followed by B and C, which is consistent with colloidal structures going from “GEL” to “SOL”.

Figure 2a and 2b: Penetration Index and Ductility – Evolution with ageing



4. EXPERIMENTAL RESULTS – COMPLEX DYNAMIC MODULUS (DSR)

The viscoelastic nature of bituminous binders can be adequately described when performing dynamic stiffness measurements under sinusoidal shear oscillation. This is done with dynamic shear rheometers (DSR) under strain amplitudes which are small enough to guarantee that the material stays within a linear domain of behaviour. The test method is now well known and covered by test standards such as EN 14770 in Europe, which has been applied in this study. The outcome of the procedure is a complex modulus, described by its magnitude (G*) and a phase angle (δ) which represents the shift between the stress and strain signals and thus quantifies the viscoelastic nature of the binder. A commonly used representation gathers the raw results obtained at different temperatures and frequencies in the form of a G* - δ plot, called a “Black diagram”. Figure 3a shows this diagram for the 3 bitumen tested after RTFOT ageing. Although all three curves tend to converge at high stiffness values reaching the glassy state, their evolution is quite different. The less the bitumen is structured (evolution from A to C), the higher its phase angle values (for a given value of stiffness) and the "quicker" its evolution towards a viscous behaviour at decreasing values of G*. Another observation to be made is related to the "smoothness" of each curve. In the case of bitumen C ("SOL" type), the overlap and continuity between results obtained at different temperatures and frequencies is almost perfect. This is less and less true for bitumen B and A. This progressive "drift away" from the time-temperature superposition principle can be ascribed to a more complex rheological behaviour associated with structural interactions.

The SUPERPAVE binder performance grade specifications [AASHTO M320-10], which are an outcome of the SHRP research program in the USA, are partly based on performance indicators built upon G* and δ. The criteria used are critical temperatures (determined at a test frequency of 10rad/s = 1,59Hz) at which G*/sinδ (associated to permanent deformation) gets below 1kPa (fresh binder) or 2,2kPa (RTFOT aged binder) and at which G*sinδ (associated to fatigue cracking and measured on the RTFOT+PAV aged binder) gets above 5 000 kPa. These critical temperatures are given in Figure 3b. The difference for G*/sinδ at high temperature between binder A and C almost reaches a value of 6°C, which is the gap between two SUPERPAVE performance grades.

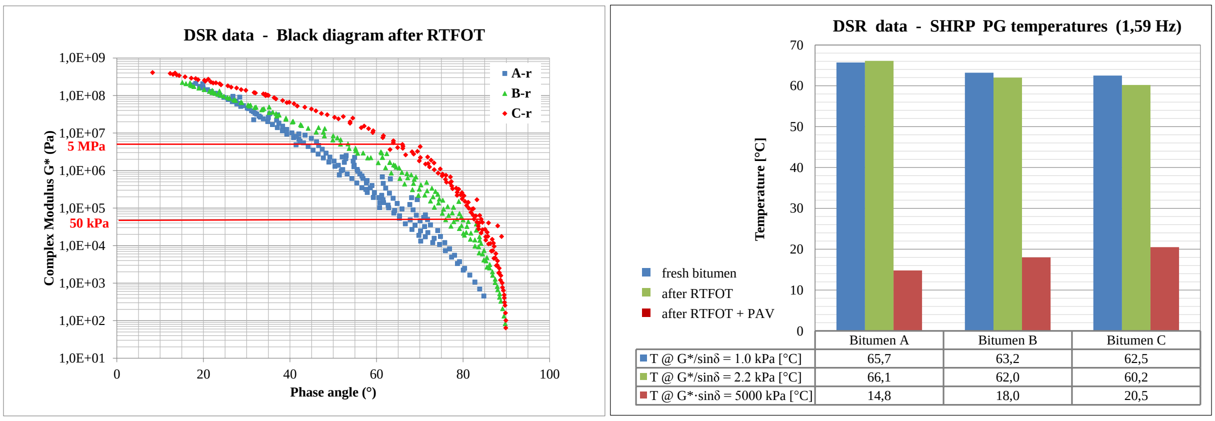


Figure 3a and 3b: DSR data – Black diagram and SHRP performance grade temperatures

There are more and more doubts about the relevance of G*/sinδ and G*sinδ, especially in the case of polymer modified binders. Such doubts may in particular be motivated by the fact that these indicators are measured in a domain of small deformations whereas permanent deformation and fatigue phenomena occur in a domain of large deformations and failure behaviour under repeated loads.
Staying at the basic level of G* and δ may nevertheless be expected to deliver an insight on binder structure, and more particularly so when looking at their evolution with temperature (temperature susceptibility) and ageing. Even if not sufficient to predict failure behaviour, this "elemental" information can certainly be of valuable help when it comes to binder selection. A possible approach is outlined hereafter:

- On RTFOT aged binders (the binder after application on the road), determine the temperatures T1 (with 8mm plate) and T2 (with 25mm plate) at which G* measured at 1,59Hz equals 5MPa (T1) and 50kPa (T2). Record the values of δ at these two temperatures. These stiffness levels have been selected so as to be measurable with rheometers complying with the minimum requirements set by the EN 14770 standard and be within the usual working ranges for 8mm and 25mm parallel plates [2].

- At these two temperatures, measure the values of G* and δ on the fresh and RTFOT + PAV aged binders so as to evaluate the sensitivity to ageing

Figure 4a shows the values of T1 and T2 and the associated values of δ for the 3 bitumen of this study. As it could be expected from the Black diagram (Figure 3a), they are clearly differentiated. Differences in temperatures T1 and T2 when going from bitumen A to bitumen C amount to around 5°C (similar to what has been established for the PG grade temperatures). The interesting additional information is however provided by the large differences observed for the values of δ.

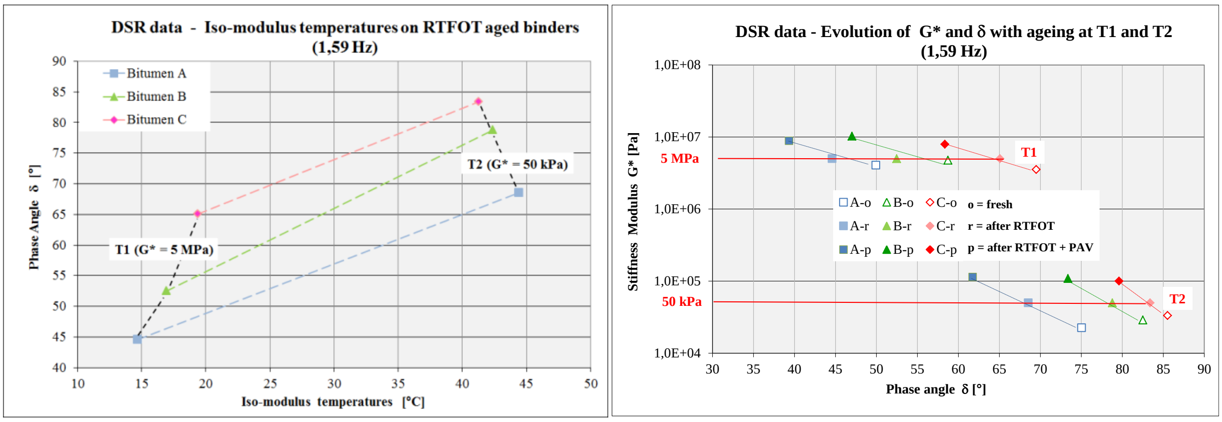


Figure 4a and 4b: DSR data – Iso-modulus temperatures and evolution with ageing

A bit surprisingly, the evolution with ageing (Figure 4b) is quite similar for the three bitumen types. Starting from a same stiffness after RTFOT, parallel lines are obtained in a G*-δ plot. It is only at the 50kPa level that we observe a tendency for these lines to become steeper (lower evolution of δ) when going from bitumen A to bitumen C. Possible reasons for these relatively small differences may be the fact that RTFOT and RTFOT + PAV ageing may still be relatively “mild” (extending PAV testing time is presently under consideration in the USA) or that the strain amplitudes applied in the linear viscoelasticity domain are too small to evidence differences in the impact of ageing. One may also wonder whether the response would have been different had the exercise been done at a different test frequency. As shown in Table 2, changing the frequency does of course modify the values of T1 and T2 but has only a limited impact on the corresponding values of δ. The same applies for the impact on the evolution of G* (expressed as a ratio to the reference values of 5MPa and 5kPa) and δ (difference with the values obtained after RTFOT) with ageing.

Table 2: Impact of test frequency on iso-modulus temperatures and evolution with ageing

		Temperature T1 [°C] G* = 5 MPa			Temperature T2 [°C] G* = 50 kPa			Value of δ at T1 [°]			Value of δ at T2 [°]		
		0,1 Hz	1,59 Hz	10 Hz	0,1 Hz	1,59 Hz	10 Hz	0,1 Hz	1,59 Hz	10 Hz	0,1 Hz	1,59 Hz	10 Hz
After RTFOT	A	6,3	14,7	21,1	31,4	44,4	54,6	43,1	44,6	47,1	66,4	68,5	71,1
	B	8,6	16,9	23,2	29,4	42,4	52,5	51,6	52,5	54,2	77,2	78,8	80,3
	C	10,4	19,4	25,8	29,3	41,3	51,6	64,4	65,1	65,1	82,7	83,4	84,3
		Ratio G*@T1 / 5 MPa			Ratio G*@T2 / 50 kPa			Evolution of δ at T1 [°]			Evolution of δ at T2 [°]		
		0,1 Hz	1,59 Hz	10 Hz	0,1 Hz	1,59 Hz	10 Hz	0,1 Hz	1,59 Hz	10 Hz	0,1 Hz	1,59 Hz	10 Hz
Fresh bitumen	A	0,77	0,81	0,83	0,38	0,45	0,49	5,5	5,4	5,1	7,3	6,5	5,5
	B	0,91	0,95	0,89	0,71	0,58	0,62	6,5	6,3	6,0	3,7	3,7	3,5
	C	0,69	0,71	0,69	0,60	0,66	0,70	4,7	4,4	4,2	2,3	2,1	2,1
		Ratio G*@T1 / 5 MPa			Ratio G*@T2 / 50 kPa			Evolution of δ at T1 [°]			Evolution of δ at T2 [°]		
		0,1 Hz	1,59 Hz	10 Hz	0,1 Hz	1,59 Hz	10 Hz	0,1 Hz	1,59 Hz	10 Hz	0,1 Hz	1,59 Hz	10 Hz
After RTFOT + PAV	A	1,80	1,77	1,71	2,60	2,27	2,00	-4,8	-5,2	-5,5	-7,2	-6,8	-6,4
	B	2,06	2,06	1,93	3,22	2,17	1,99	-5,2	-5,5	-5,7	-6,2	-5,4	-4,6
	C	1,60	1,59	1,46	2,17	2,00	1,86	-6,7	-6,7	-7,5	-4,0	-3,8	-3,5

5. EXPERIMENTAL RESULTS – MSCRT (DSR)

The Multiple Stress Creep Recovery Test (MSCRT) has been developed so as to get a better insight on the permanent deformation behaviour of bituminous binders at high temperatures under repeated shear load conditions and in relation to level of stress [AASHTO TP70-12, prEN 16659]. The test is run on a DSR apparatus, with a 25mm parallel plate and 1mm gap setting. At a given stress level, 10 loading cycles are applied. At each cycle, the sample is loaded for 1s at constant stress, and then allowed to recover for 9s. After each sequence of 10 loading cycles, two specific characteristics are determined:

- The % recovery, which is the average of the recovered deformation recorded at the end of each individual loading cycle.
- The non-recoverable creep compliance (Jnr), which is the average of the ratio (non-recovered strain/applied stress) calculated at the end of each individual loading cycle. The lower the Jnr value, the less the binder should be prone to rutting.

In the frame of this study, the MSCRT was run at 60°C and the "standard" creep stress values of 0,1kPa and 3,2kPa complemented with a third higher level of 6,4kPa [3]. The results are shown in Figures 5a and 5b. As expected, Jnr values are lowest for the most structured bitumen (bitumen A) which is also the only one showing some elastic recovery behaviour. Bitumen A appears further to be the most sensitive to the applied level of stress. It is also quite interesting to notice that the impact of ageing as measured by the MSCRT (diminution of Jnr) seems to be more and more pronounced when going over from a "SOL" (bitumen C) to a "GEL" structure (bitumen A). This is consistent with gel features: under oxidation, more polar functions are formed increasing molecular interactions and making "a gel become more gel".

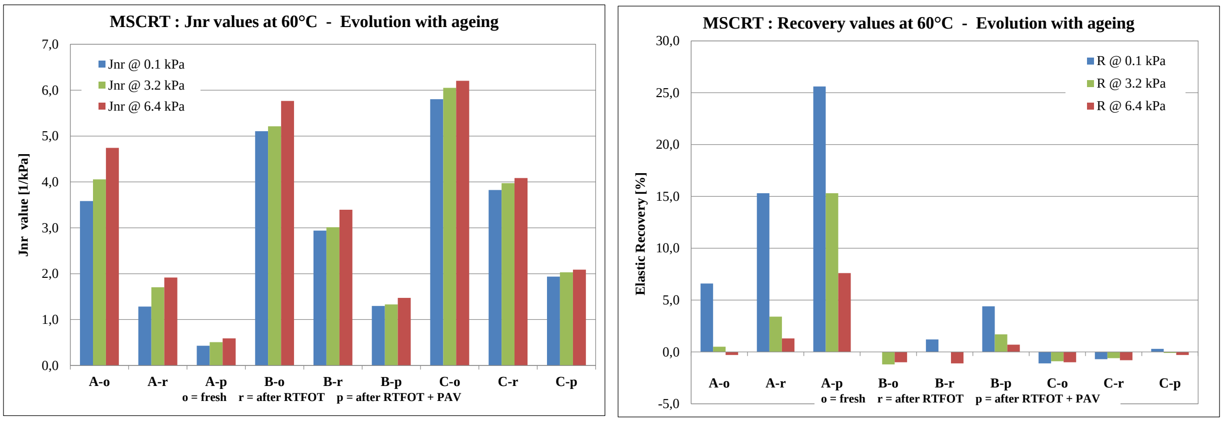


Figure 5a and 5b: MSCRT data – Evolution with stress level and ageing

6. EXPERIMENTAL RESULTS – LOW TEMPERATURE BEHAVIOUR

6.1 Test methods

Low temperature behaviour has been assessed via three different methods which are briefly reminded or described hereafter. In each case, testing has been done on the fresh, RTFOT and RTFOT+PAV aged binders.

The **Fraass test** [EN 12593] is performed on a thin film of binder (0,5mm) coated on a thin metal plate which is cooled down at a fast rate (**1°C/min**) and repeatedly flexed until failure is obtained (Fraass breaking point temperature). The procedure has the advantage of characterizing failure behaviour but is often blamed for poor reproducibility, however more so for polymer modified binders than for pure bitumen. In the frame of this study, automatic BPA 5 equipment has been used and all tests have been performed by a single laboratory so as to escape reproducibility artefacts.

The **Bending Beam Rheometer (BBR)** [EN 14771], another SHRP outcome, characterizes flexural creep stiffness at low temperature while applying a constant load (100g) on a beam sample (127 x 12,7 x 6,4mm) for 240s. At a loading time of 60s, two characteristic temperatures are determined from this test:

- $T_{S=300\ MPa}$, which is the temperature at which stiffness becomes equal to 300MPa
- $T_{m=0,3}$, which is the temperature at which the slope m (absolute value) of the log(S) versus log(time) curve becomes equal to 0,3. The m-value can be seen as indicating the capability of the material to relax under an applied stress.

It is to be mentioned that, contrarily to Fraass which is a failure test, the BBR procedure characterizes stiffness and relaxation in the linear domain. A relationship to failure is then a matter of correlation, which is most likely not universal (i.e. not equally applicable to all kinds of binders).

The **ABCD (Asphalt Binder Cracking Device)** test [AASHTO TP92-11] has been developed in the USA as the binder counterpart of the TSRST (Tensile Strength Restrained Specimen Test) procedure on bituminous mixtures. The bituminous binder is poured into a silica mould around an invar steel ring (outer $\Phi \approx 54$mm). The cross-sectional area of the binder is 0,5" x 0,25" (12,7 x 6,35mm). The mould is then placed in an environmental chamber and cooled down at a fixed rate (20°C/h starting from 20°C in the case of AASHTO TP92-11). As it tends to shrink, the binder compresses the invar ring and the resulting stress is obtained from strain gauges mounted on the invar rings. The location of the failure is controlled via a protrusion (reduction of cross-sectional area) placed at a given point of the circumference. Theoretical considerations (stress concentration factor) [4] allow calculating the actual fracture stress at the protrusion from the recorded average thermal stress at failure.

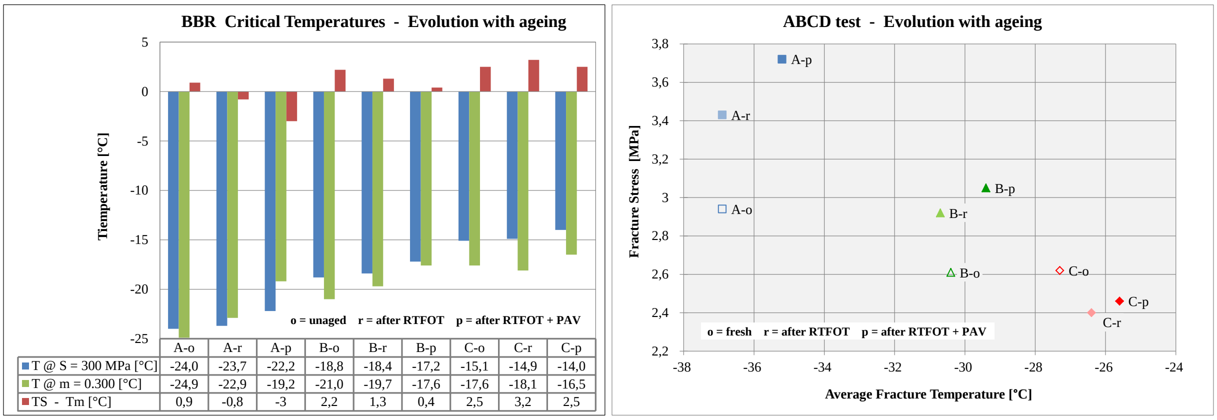
Figure 6: ABCD test equipment and samples

For this study, the ABCD tests have been contracted to the Western Research Institute (WRI, Laramie, WY, USA). To be as close as possible to the practice of the TSRST test on bituminous mixtures, the starting test temperature has been set at 5°C and the cooling rate slowed down to **10°C/h.**

6.2 Test results

The comparison of the outcome of the BBR and ABCD tests is done in Figure 7. The critical BBR temperatures become more favourable when going from the "SOL" to the "GEL" type of bitumen. $T_{m=0,3}$ is generally lower than $T_{S=300\ MPa}$. This difference is more marked for bitumen C and gets lower for bitumen B and especially A. Also the impact of ageing seems to be related to binder structure, the evolution being less favourable for the most structured bitumen. This is best illustrated by the $T_{m=0,3}$ value which, for bitumen A, becomes even higher than $T_{S=300\ MPa}$ after RTFOT + PAV. Conversely, the $T_{S=300\ MPa}$ temperature seems to be much less sensitive, the impact of ageing being almost nil after RTFOT and only small after RTFOT+PAV. These results confirm the general trend that binders become more m-controlled after RTFOT+PAV ageing.

The ABCD test shows larger differences in fracture temperature, clearly to the advantage of the most structured bitumen which are also able to withstand higher stresses. For bitumen A and B, RTFOT ageing leads to higher failure stresses but does not affect fracture temperature. For these two binders, the fracture stress continues to increase after RTFOT + PAV while the fracture temperature is only slightly reduced. In comparison, and contrarily to what would be suggested by the BBR results, bitumen C seems to be more affected by ageing.



	A-o	A-r	A-p	B-o	B-r	B-p	C-o	C-r	C-p
T @ S = 300 MPa [°C]	-24,0	-23,7	-22,2	-18,8	-18,4	-17,2	-15,1	-14,9	-14,0
T @ m = 0.300 [°C]	-24,9	-22,9	-19,2	-21,0	-19,7	-17,6	-17,6	-18,1	-16,5
TS - Tm [°C]	0,9	-0,8	-3	2,2	1,3	0,4	2,5	3,2	2,5

Figure 7a and 7b: Comparison of BBR and ABCD data – Evolution with ageing

The ABCD fracture temperatures are well in line with the BBR critical temperatures. However, the correlation appears to be stronger with $T_{S=300\ MPa}$ than with $T_{m=0,3}$ (Figure 8).

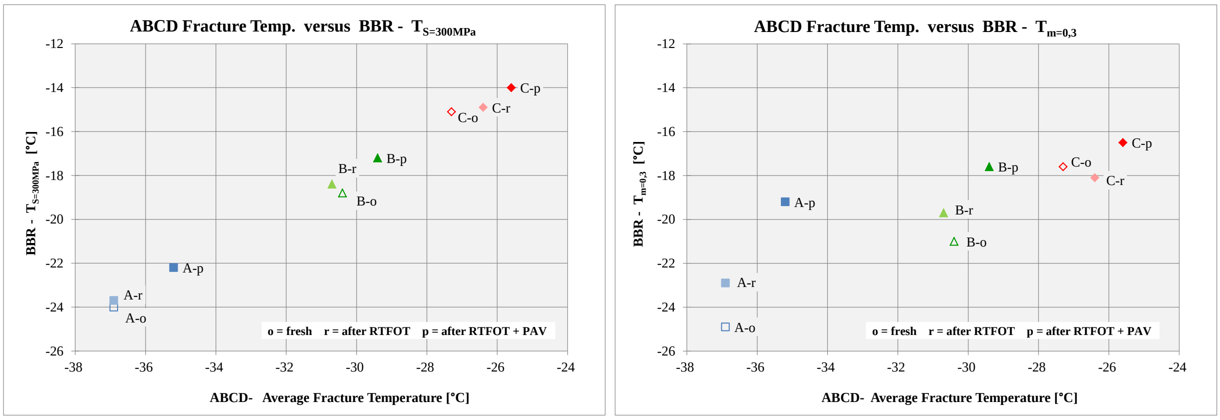


Figure 8a and 8b: Relationship between ABCD Fracture Temperature and BBR critical temperatures

Although both tests measure a failure behaviour, the correlation between ABCD Fracture Temperature and Fraass Breaking Point is not perfect (Figure 9a). A quite good relationship is however observed between Fraass Breaking Point and $T_{m=0,3}$ (Figure 9b). These findings suggest that the ABCD test, in which the rate of temperature decrease is relatively low, would be more specifically governed by strength (Figure 8a) whereas the Fraass test, much more severe in terms of imposed cooling rate and imposed deformations, would rather be governed by the binder relaxation abilities as seen by the m-value. The difference in cross-sectional sample area between the two tests may also be part of the explanation.

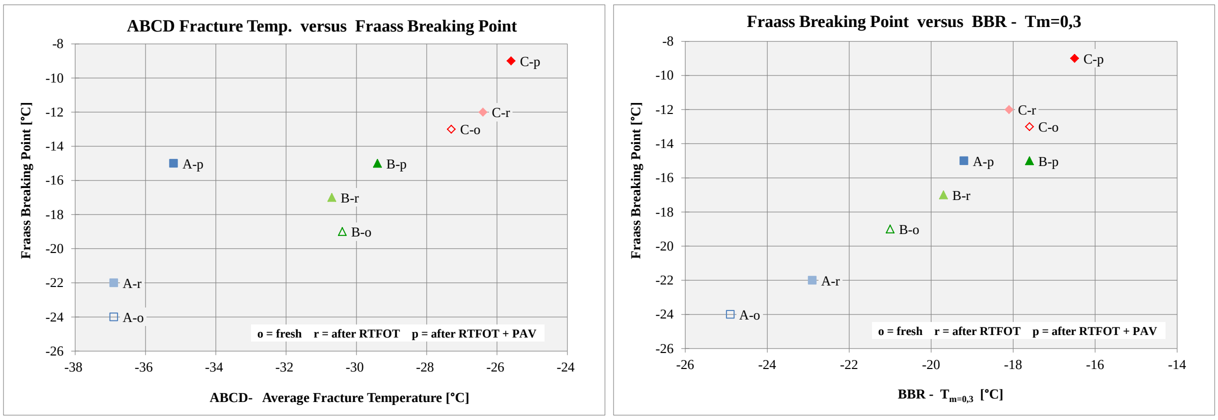


Figure 9a and 9b: Relationship between failure tests and BBR critical temperatures

7. EXPERIMENTAL RESULTS – LAS TEST WITH DSR EQUIPMENT

7.1 Description of the test method

The Linear Amplitude Sweep (LAS) test was developed by the Modified Asphalt Research Center (MARC) at the University of Wisconsin. It has been proposed as an alternative to the time sweep test (which is performed at a constant strain level) to evaluate binder fatigue resistance. Since its first adoption by AASHTO [AASHTO TP 101-12] the method has been revised by MARC and the most recent version, which has been used by WRI in this study, is TP 101-12-UL [5].

Since it is intended to predict fatigue resistance, the test has to be run at a temperature which is sufficiently low to eliminate flow behaviour, yet not too low to prevent adhesive failure between the binder sample and the DSR plates. The allowable range of the complex shear modulus G* is estimated to be 10 to 50MPa [6]. A G* value of **30MPa** was selected for this study as a compromise.

There are four main steps in performing the LAS test according to TP 101-12-UL:

- Determination of the iso-modulus temperature at a frequency of 10 Hz.
- Performance of a frequency sweep (0,1 to 30Hz) at 0,1% strain at the iso-modulus temperature.
- Performance of a strain ramp function at 10Hz and at the iso-modulus temperature. The strain is increased from 0,1 to 30% over a total of 310s (3 100 cycles).
- Analysis of the frequency sweep and strain ramp results to calculate a theoretical fatigue line on the basis of viscoelastic continuum damage (VECD) theory [5, 6, 7]. The procedure is schematically outlined in Figure 10.

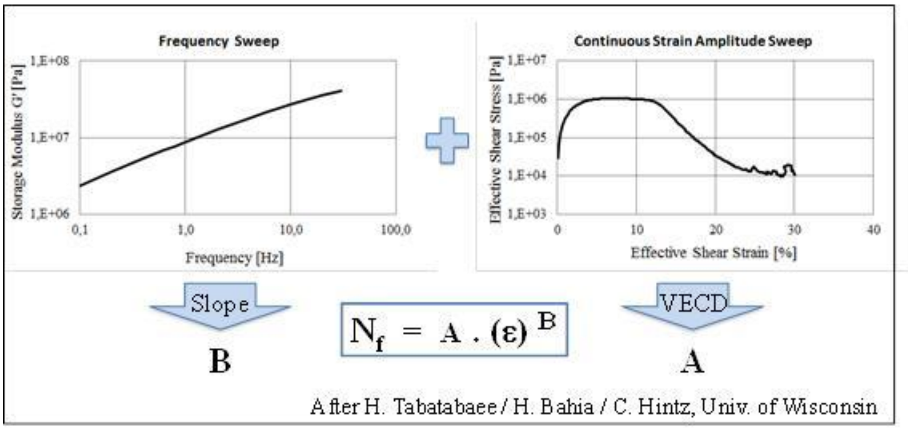


Figure 10: Determination of Fatigue Life from LAS test results

7.2 Test results

The iso-modulus (G* = 30MPa) temperatures at 10Hz for the different bitumen are given in Figure 11a and the "VECD fatigue lines" obtained after RTFOT ageing are shown in Figure 11b. These lines reveal a greater sensitivity to strain level (steeper slope) for the most structured bitumen. They may be considered as being relatively close but it should not be forgotten that this "intrinsic" behaviour is observed at about 15°C for bitumen C while it only occurs at about 7,5°C for bitumen A. A bit surprisingly, the evolution with ageing, which is shown in Figure 12 for bitumen A and C (B showing an intermediate behaviour), is very small and the number of life cycles tends even to improve at lower strains. This would show that RTFOT and RTFOT+PAV ageing have very little impact on the "intrinsic" damage mechanism as evaluated through the LAS procedure under iso-modulus conditions. RTFOT and RTFOT+PAV ageing do however impact the corresponding iso-modulus temperatures (these temperatures increase with ageing).

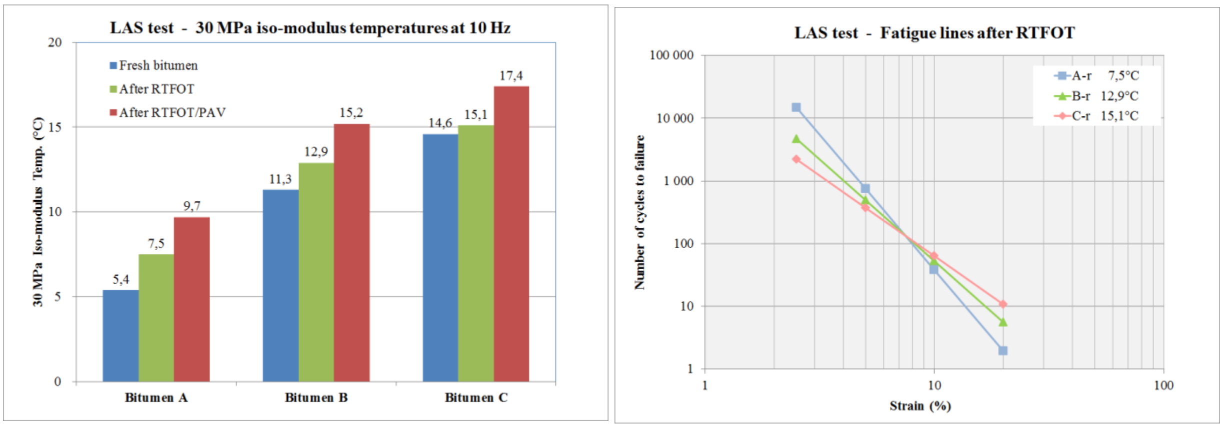


Figure 11a and 11b: LAS test – Iso-modulus temperatures and fatigue lines after RTFOT ageing

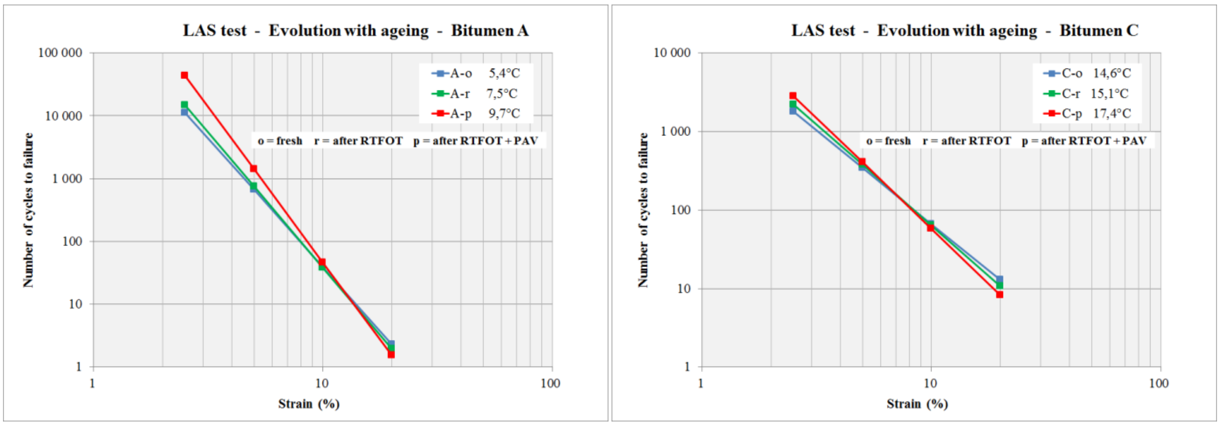


Figure 12a and 12b: LAS test – Evolution of fatigue lines with ageing

In the domain of high stresses and strains, the corresponding signals tend to become distorted. One may then question the validity of the determined G* and δ values, and hence of the damage calculation through VECD theory. This point is specifically addressed in [7].

A more elemental analysis can however be done by simply looking at the shape of the stress versus strain curve. As shown in Figure 13, one may identify a "critical strain" value above which stress starts to decrease whereas δ flattens-up and starts to increase again. This is where the material starts to undergo unrecoverable damage (as we could verify from a few hysteresis tests conducted before and after this limit).

Figure 14 shows the impact of ageing on the development of stress under iso-modulus conditions for bitumen A and C. The analysis confirms that aged bitumen may exhibit a better "intrinsic" resistance to increasing strains than the corresponding fresh bitumen. This is more particularly true for the most structured bitumen while the intrinsic behaviour seems not really altered for bitumen C.

A small investigation has also been made on the impact of test temperature for bitumen A and C. In the contemplated range, and for both bitumen, the impact on the critical strain proved to be rather small (Figure 15). It is however interesting to notice, especially for bitumen C-o, that reducing the temperature from 14,6°C to 5,4°C does also modify the slope of the stress curve after the critical strain. It tends to become steeper and stress values become erratic more rapidly. Another observation, which confirms the outcome of the VECD approach, is that under iso-modulus conditions (5,4°C for bitumen A-o and 14,6°C for bitumen C-o), the stress curve is more favourable for bitumen A-o in its first part (higher critical strain value) but tends to collapse more rapidly in the second part (higher strain levels). A similar exercise conducted on bitumen A and C after RTFOT+PAV ageing led to the same observations.

Further considerations on non-linearity testing and its relationship to fatigue are to be found in [8].

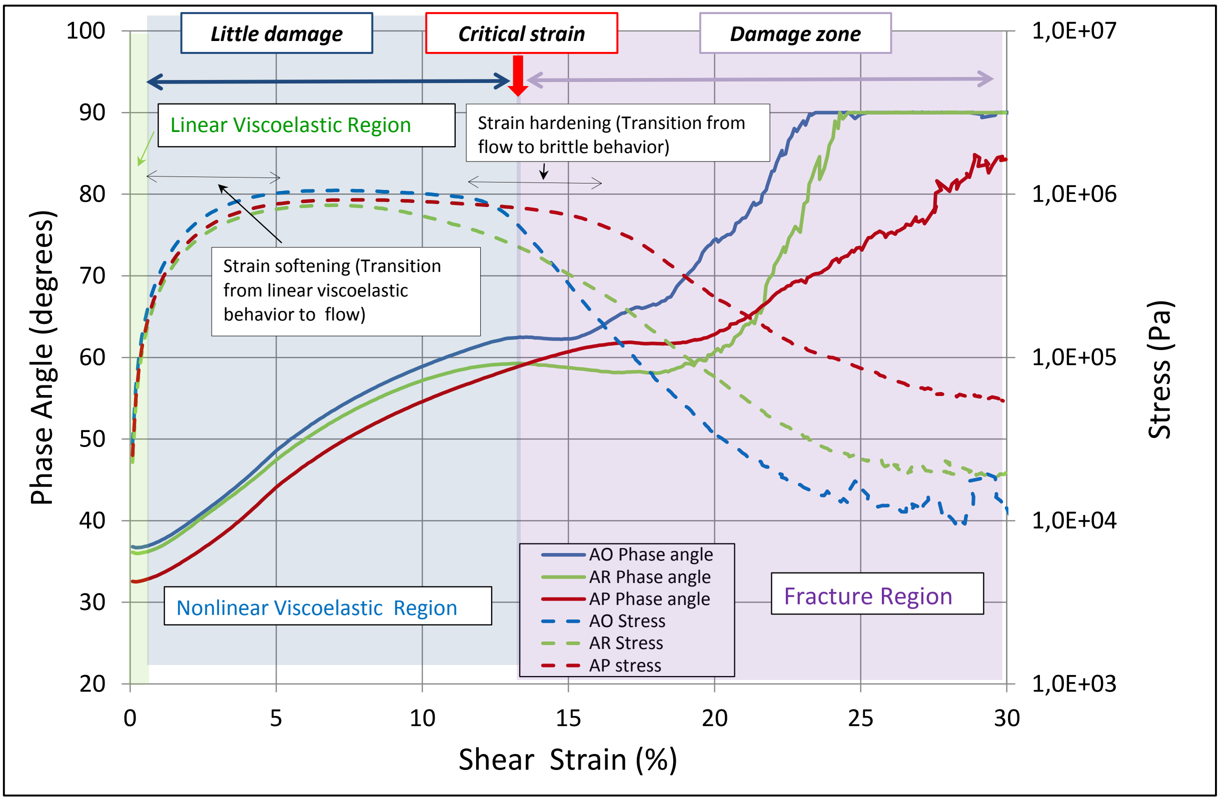


Figure 13: Evolution of stress and δ with increasing strain in the LAS test

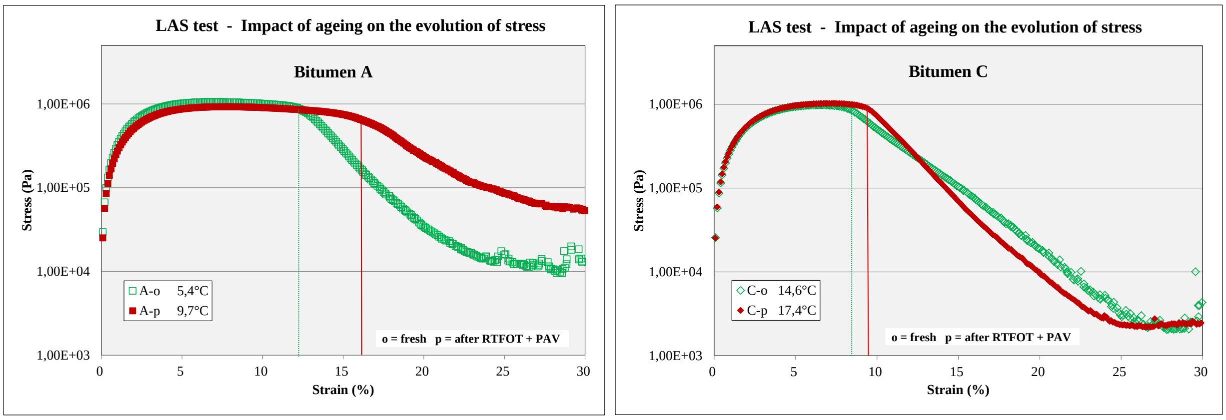


Figure 14a and 14b: LAS test – Impact of ageing on the stress vs strain curve

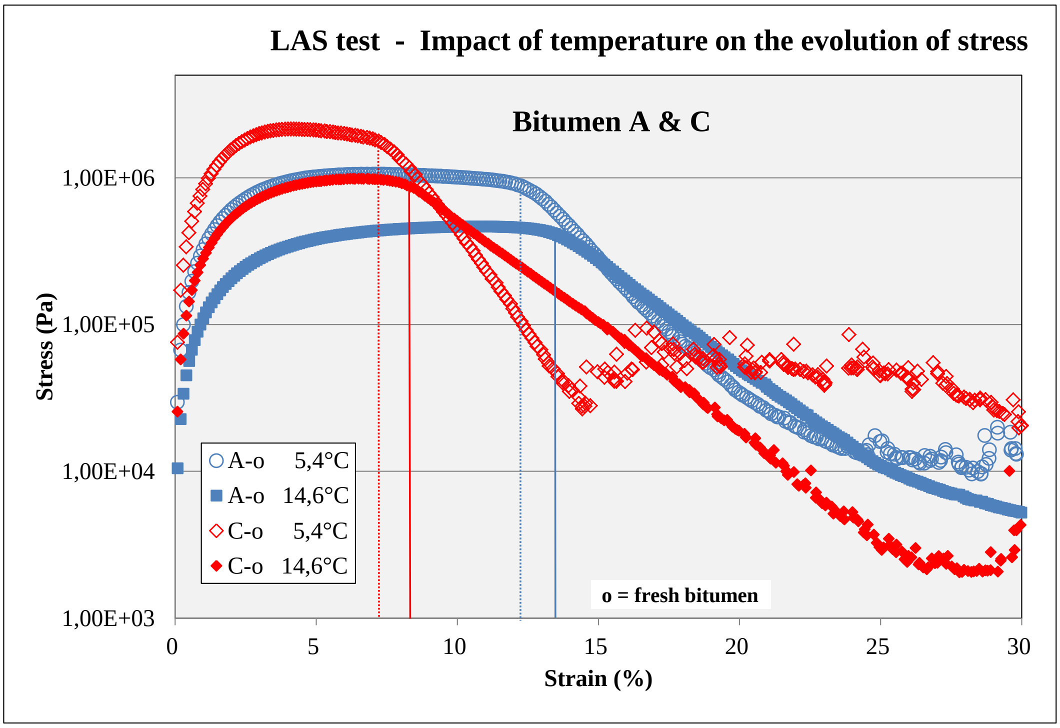


Figure 15: LAS test – Impact of temperature on the stress vs strain curve

8. CONCLUSIONS

Coming back to the question at the origin of this study (ability of different test methods to differentiate binders), we may summarize our finding by a number of tentative statements.

- In the case of pure bitumen, conventional properties clearly keep a definite value, especially when considering indicators related to sensitivity to temperature and ageing. Concerns about their precision (Penetration index, Fraass breaking point) and relevance, which are however more justified for modified binders than for pure bitumen, should not prevent from using them.
- By comparison, DSR data (G* and δ) measured in the linear domain of viscoelasticity offer the great advantage of measuring fundamental properties and, in particular, of adding information on the phase angle. The fact that, in our study, G* and δ were not really able to evidence differences in ageing behaviour in relation to bitumen structure has to be further investigated on a wider range of products.
- The MSCRT procedure was originally intended for polymer modified binders but it delivers also some good insight into bitumen structure when considering its dependency upon the applied level of stress.
- Flexural creep stiffness and its sensitivity to loading time, as measured by the BBR test, is not directly related to failure behaviour. Nevertheless, in the case of pure bitumen, correlations may probably be established. As suggested by the examples of Fraass and ABCD, one or the other of the $T_{S=300\ MPa}$ or $T_{m=0,3}$ parameters may be more appropriate depending on how the binder is actually stressed in service. From a practical point of view, this suggests that both parameters should be kept in the frame of a bitumen evaluation scheme.
- The LAS test, although its conditions and interpretation through VECD theory and relationship to fatigue will certainly continue to be debated, has the great advantage of being a fast test for the assessment of a material’s response to increasing strain amplitudes. Working at a fixed level of stiffness allows comparing intrinsic behaviour. In our study, intrinsic “VECD fatigue” (at iso-modulus temperature) did not appear to be highly affected by ageing, which suggests that the essential part of sensitivity to ageing could be “captured” by the evolution of stiffness. Another approach could be to describe the evolution with temperature of the LAS stress/strain curves. This may help to approach the

high stiffness levels at which binders work in actual fatigue tests on bituminous mixtures (e.g. 10°C–25Hz) but may be limited by the capabilities of DSR rheometers.
- The results obtained here on a relatively "soft" bitumen grade should now be conducted in a similar way on harder grades, in particular to confirm the sensitivity of the different test methods to ageing.

Overall, our results illustrate once more the strong dependency of bitumen response on the applied loading conditions. Bituminous binders need to be looked at from different angles. Only experience, but also sound engineering judgement, will tell how the resulting picture relates to actual performance on the road. But it is better to work with good pictures rather than with rough sketches.

REFERENCES

[1] *Contribution à la connaissance des propriétés des bitumes routiers*, C. Gaestel, R.Smadja K.A. Lammiman, Rev. Gen. Routes et Aérodromes, 466, 85 (1971)

[2] EN 14770:2012 –*Bitumen and bituminous binders – Determination of complex shear modulus and phase angle – Dynamic Shear Rheometer (DSR)*

[3] *New developments in predicting rutting of asphalt mixtures from binder rheological characteristics*, J. P. Planche, 16th IRF World Meeting, Lisbon, 2010.

[4] *Asphalt Binder Cracking Device to Reduce Low-Temperature Asphalt Pavement Cracking*, S. Kim, Final Report, FHWA-HIF-11-029.

[5] AASHTO TP 101-12-UL, *Estimating Damage Tolerance of Asphalt Binders Using the Linear Amplitude Sweep*), available on the MARC website: http://uwmarc.wisc.edu/linear-amplitude-sweep

[6] *Simplification of the Linear Amplitude Sweep (LAS) Test and Specification Parameter*, C. Hintz and H. Bahia, Transportation Research Board Annual Meeting January 2013, Washington, D.C.

[7] *Nonlinear oscillatory shear tests applied to bitumen: analysis and application of large amplitude oscillatory shear (LAOS),* A.O. Cookman, M.J. Farrar, J.P. Planche, 6th Eurasphalt & Eurobitume Congress, Prague 2016.

[8] *Linearity in Bituminous Materials During Cyclic Tests,* G. Gauthier, D. Bodin, E. Chailleux, T. Gallet, Int. Journal on Road Materials and Pavement Design, 2010 EATA special issue – June 15-16th 2010, Parma