Evaluation of performance tests on bitumens

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ABSTRACT

The trend in modern pavements is moving towards bitumens with higher performance for longer term service in roads. There is a demand for better strength and adhesion, lesser road maintenance and road closures. It can be observed large increase in traffic load and application of new techniques which require more innovative binders. Hand in hand with mentioned changes new material evaluation is taking place. Bitumen properties are more often characterized in relation to temperature, loading time, frequency, stress or deformation. One of the base flow techniques is a repeated creep that is measured with dynamic shear rheometer as a tool of study of bitumen viscoelastic properties. The method is under investigation currently and many road agencies worldwide applied the results in to the specifications.
This paper deals of the use of dynamic mechanical analysis for better bitumen characterization at selected polymer modified bitumens as well as the evaluation of Multiple Stress Creep and Recovery Test based on proposed prEN 16659 standard.

Keywords: Asphalt, Creep, Modified Binders, Performance testing, Rheology

1. INTRODUCTION

Bituminous binders are complex materials with its defined colloidal structure. The nature of bitumen determines basic mechanical characteristic which are not easily understood as other building materials. It is due to the fact that bituminous materials exhibit partly viscous and partly elastic behavior. The rheology of the bitumen at a given temperature is determined by both the constitution and structure of the hydrocarbons and heterocyclics. Chemical composition is more complex, thus instead of extremely laborious analysis it is better in advance to divide bitumen into four chemical groups called asphaltenes, resins, aromatic oils and saturated oils. This division enables bitumen to compare its rheology with composition. Changes in composition and structure can result in change of the rheological behavior. Different stresses / strains, temperatures, rate of stresses /strains, film thicknesses and material history play important role in evaluating basic material properties including rheological ones. It is important to characterize the viscoelastic behavior to better understanding their significance in real life performance. This is one of the objectives of this paper. Further, the goal is to find a good technique to reveal the differences among materials currently used to modify base bitumen.

Typically, about 14 percent of total bitumen consumptions in European Union are taken by polymer modified bitumen (PMB) [1]. There are several reasons why bitumen modification is gaining its attention. It could be by demand for better strength and adhesion, lesser road maintenance, increase in traffic, changes in base bitumen or application of new materials and techniques which require more resistant materials. The main causes of road failure are permanent deformation (rutting), fatigue cracking, thermal cracking and moisture damage. Polymer modified bitumen is used to prevent these modes of failure, which according to the Association of Modified Asphalt Producers (AMAP), occur in the following proportion: 83% rutting, 43% fatigue cracking, 39% thermal cracking and 9% moisture damage (stripping) and others [2]. Even though the PMB results from conventional tests as Softening Point, Penetration, Elastic Recovery and Force Ductility, etc. significantly differs from base bitumen due to fact that PMB has different rate of viscous and elastic behavior.

2. RHEOMETER METHODS

2.1 The role of specifications

European Asphalt Pavement Association (EAPA) initiated and CEN/TC227 issued second generation of asphalt standards in Europe. The goals were to create performance oriented set of specifications dealing with asphalt mix in the back of the truck. As a logical step, new bituminous specifications shall follow this trend as well as did it in North America several decades ago. This effort led to establish grading system based on rutting and fatigue while using artificially aged bitumen to evaluate high temperature material behaviour. The key parameters denoted as Complex Shear Modulus (G^*) and Phase Angle (δ), start to play the critical role in terms of bitumen specification. This can be translated as material stiffness and / or resistance to deformation either permanent or recoverable. The Performance Grade (PG) system has replaced the viscosity and penetration grading systems in the U.S. [2]. This is also due to the fact that modern equipments enable to measure given functions relatively fast, at reasonable repeatability and reproducibility with easy and understandable output.

Multiple Stress Creep and Recovery Test (prEN 16659) was found to be the best rheological test for correlation of bituminous binders to asphalt mixture behaviour. The need of performance related specification is becoming more important in terms of bitumen sustainability. In some cases, due the inconsistency of the bitumen during the production and more ever to distinguish among many additives and polymers recently used, shear tests become a useful tool how to find real material properties. The recent trend is to evaluate different products (polymers and additive) available on the market, with the best possible performance related to the mechanical testing.

2.2 Oscillations

The Absolute value of Complex Shear Modulus and the Phase Angle, which describes the ratio between the shear stress and shear strain, are known to the industry for many decades [4]. Most currently, the procedure is well defined at AASHTO and EN specifications [2,4]. Performance specifications define a single point by dividing $|G^*|$ by $\sin \delta$. This parameter describes how is the material characterized from the temperature at which this falls at value of 1000 Pa. The complex modulus is determined by placing a sinusoidal shear stress of amplitude σ_o (Pa) and angular frequency ω (Hz) on the sample and measuring the resulting shear strain γ_o (-). It is believed that all measurements are conducted in linear viscoelastic range that characterizes the independency on amplitude. The U.S. PG specification considers one loading frequency of 1.59Hz [6]. The advantage is to measure given materials at numerous frequencies, which may represent a better material picture. Up to the point this is the case of EN standard evaluating bitumen at a frequency range at temperature of 60 °C. In terms of energy, the strain may be either dissipated as heat or stored elastically. These responses are contained in the phase angle as the loss tangent ($\tan \delta$), being proportional to the ratio of energy dissipated to the energy stored per cycle. There are some studies showing advantages of using multiple temperatures and frequency loadings to completely characterize the bitumen. This complex phenomena is using the principle of Time Temperature Superposition (TTS) [8]. In this case, DSR testing range is extended to decades proving a full scale picture of the material behaviour, **figure 1**.

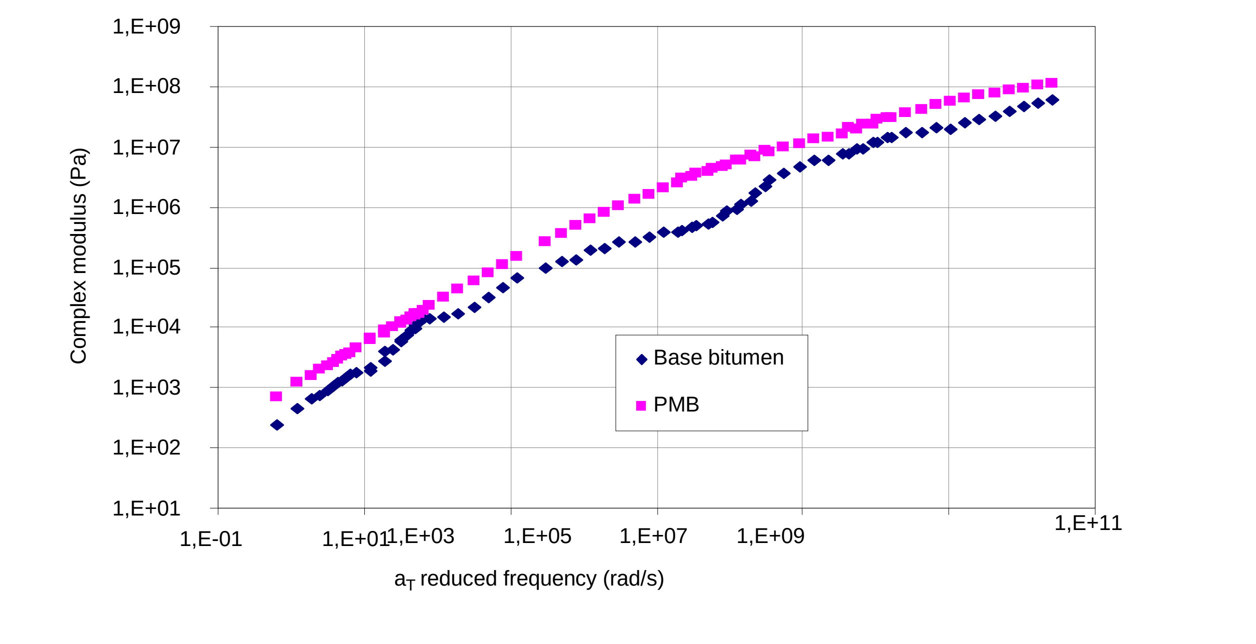

Figure 1: differences between PMB and base bitumen using TTS [9]

2.3 Creep testing
The trend in last decade is to evaluate materials in terms of repeated loadings. The creep and recovery protocol was successfully adopted and each binder at evaluated at stress levels of 100 Pa and 3200 Pa respectively, **figure 2**.

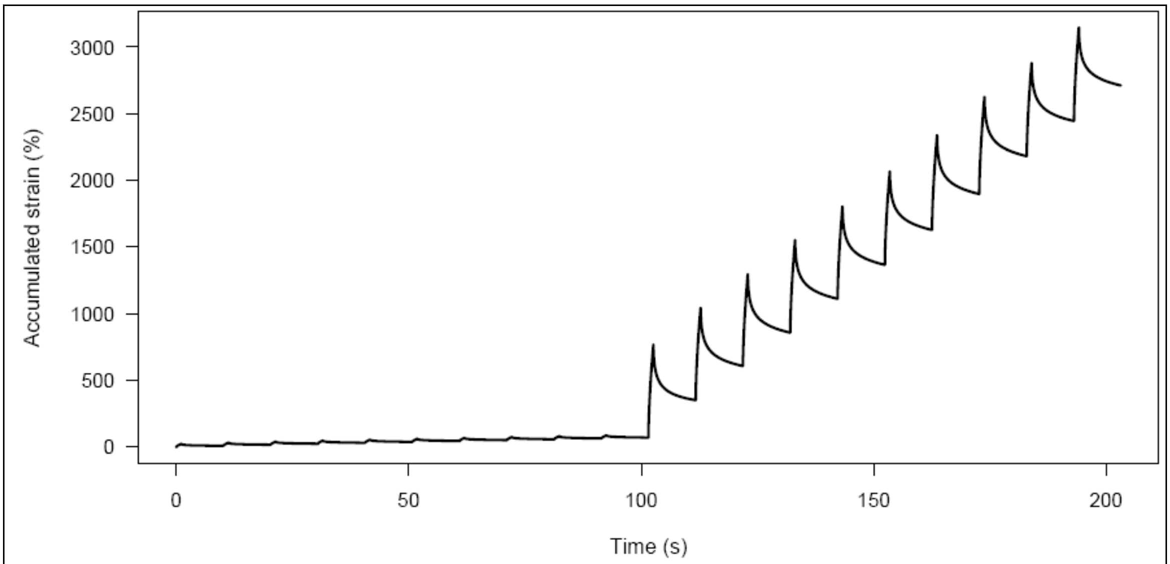

Figure 2: prEN 16659 procedure, example of MSCR test at temperature 76 °C, SBS+RET polymer [3]

3. EXPERIMENTAL

3.1 Materials used

Eight polymer modified binders were selected for this study. The modification was done with two different polymers – Styrene Butadiene Styrene (SBS) that creates mechanical bonding; Reactive Elastomeric Terpolymer (RET) that chemically reacts with bitumen and Crumb rubber polymer modified bitumen (CRmB). Some of the specimens are commercial grades used across the continent. CRmB is commercial terminal blended type with medium content of fine crumb rubber giving lower viscosity and with additive improving compatibility and storage stability.
In several cases laboratory specimens were blended with base bitumens and homogenized till the reaction ended. Each quantity sufficient to prepare sets of samples.

For the production of chemically cross-linked PMB’s, correct choice of the basic bitumen is important. RET polymers contains an epoxy groups which react with the asphaltenes and bind them with the covalent bond. Lack of asphaltenes or relative abundance of the polymer can lead to low efficiency of these reactions or reactions to each other with polymer molecules. For this reason it is important to pay attention to the chemical composition of the base bitumen, in most cases, is the typical semi-blown 50/70 pen bitumen used. Four samples were compared – base bitumen and three PMB´s prepared with SBS, RET and / or PPA (Polyphosporic Acid) modifications. Where only PMB sample no.7 was sulfur cross-linked. The polymer content for SBS varies between 3~5 percent, combination of SBS and RET was between 1~2 percent for SBS, respectively 1~2 percent for RET. PMB with RET alone contains up to 0.2 percent PPA. Marking as well as polymers and conventional properties are presented in table 1.

3.2 Testing procedures

The base binders were heated at temperatures between 180 – 185 °C and polymers were added, homogenized by laboratory high shear mill, till the reaction ended. Each quantity sufficient to prepare sets of samples. Test results are collected in following tables and figures, where Critical Temperature means temperature when G*/sinδ =1kPa, table 2, respectively G*/sinδ =2.2kPa, table 3.

Table 1: Evaluated materials

Sample no.	PMB Grade	Modifier Type	Softening Point (°C)	Elastic recovery at 25 °C (%)	Penetration at 25 °C (mm^{-1})
1	25/55-60	SBS	66.0	86	39
2	45/80-65	RET+SBS	66.3	86	46
3	25/55-60	RET	60.4	67,5	46
4	25/55-60	RET+SBS	62.0	64	46
5	45/80-60	RET+SBS	65.8	80	64
6	45/80-60	RET	60.2	80	63
7	65/105-75	SBS	90.3	100	73
8	30/70-60	CRmB	64.6	71	36

Table 2: Results of PG grading and Jnr for given materials unaged material

Sample no.	PG	Jnr (tested at PG)		Critical Temperature (°C)	Jnr (tested at critical temperature)	
		0.1 kPa^{-1}	3.2 kPa^{-1}		0.1 kPa^{-1}	3.2 kPa^{-1}
1	76	5.74	8.69	77.7	7.02	10.90
2	76	3.74	5.10	80.1	5.88	8.39
3	76	3.73	5.60	81.4	6.95	10.62
4	76	3.82	5.27	81.9	7.38	10.48
5	76	1.70	2.25	81.9	3.38	5.24
6	76	3.36	5.38	79.1	4.76	7.94
7	76	0.15	5.27	78.8	0.29	6.39
8	82	3.74	8.11	87.8	5.75	14.64

Table 3: Results of Jnr test for given materials after RTFO

Sample no.	PG	Jnr (at PG)		Critical Temperature (°C)	Jnr (at critical temperature)	
		0.1 kPa^{-1}	3.2 kPa^{-1}		0.1 kPa^{-1}	3.2 kPa^{-1}
1	70	1.88	2.62	73.8	3.09	4.46
2	76	1.72	2.27	76.8	1.84	2.52
3	76	1.69	2.49	79.2	2.53	3.96
4	76	1.65	2.29	80.3	2.80	4.04
5	76	0.71	0.83	78.9	1.03	1.31
6	76	1.37	2.00	76.9	1.53	2.28
7	70	0.17*	1.50*	73.4	0.08	0.49
8	88	0.45*	1.13*	89.9	1.45	5.99

***** tested at temperature 76 °C

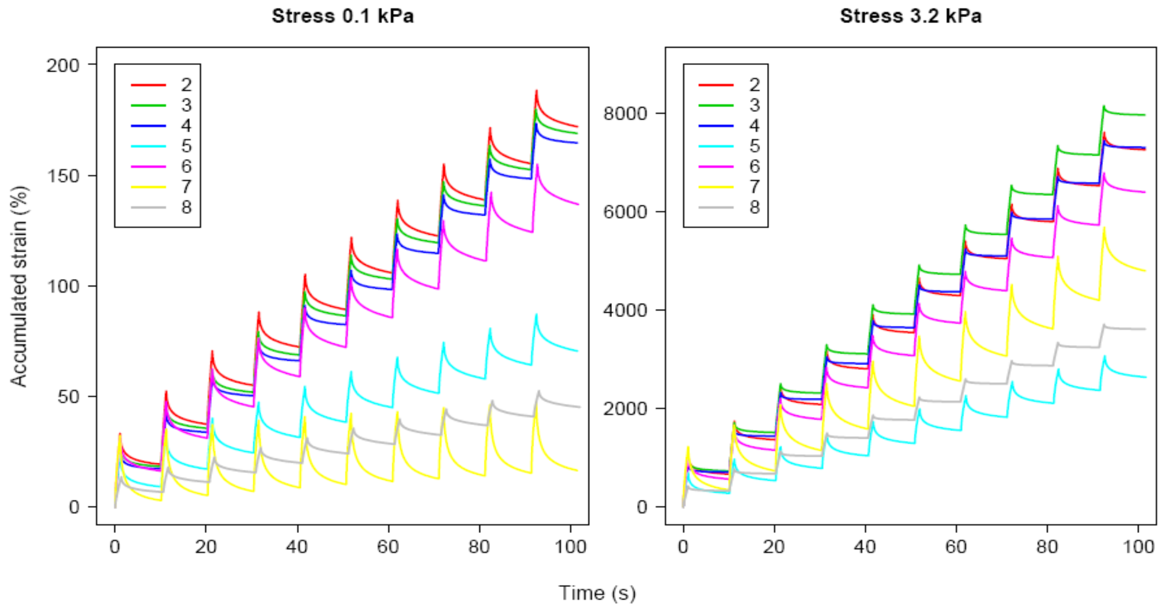

Figure 3: Multiple Stress Creep and Recovery test, original modified bitumens, at temperature 76 °C

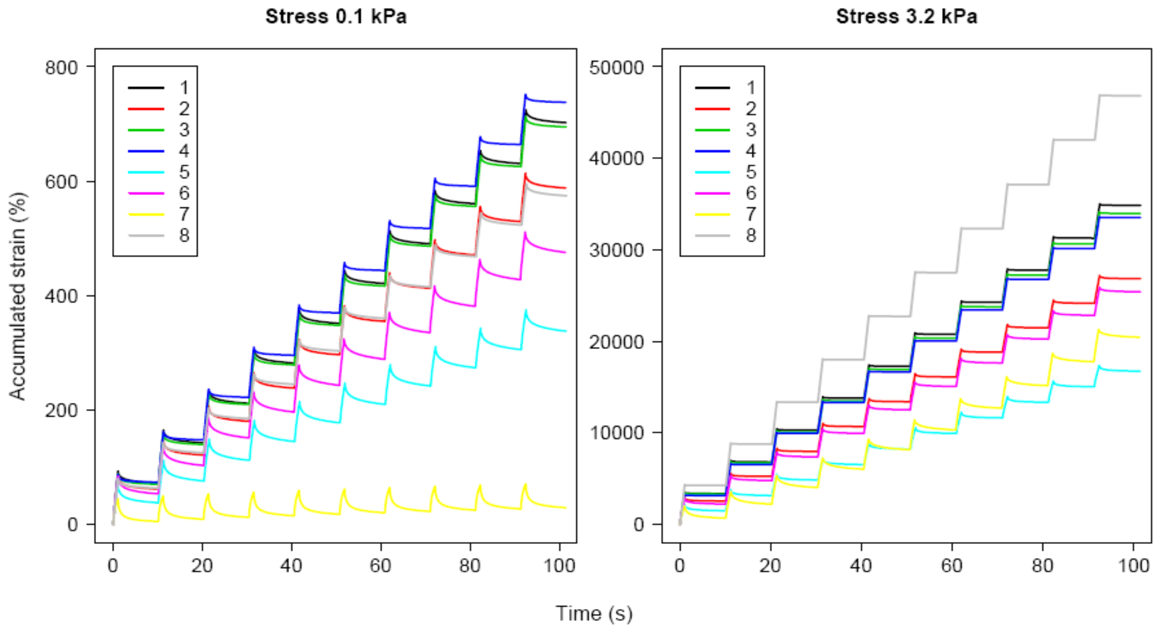

Figure 4: Multiple Stress Creep and Recovery test, original modified bitumens, at critical temperatures

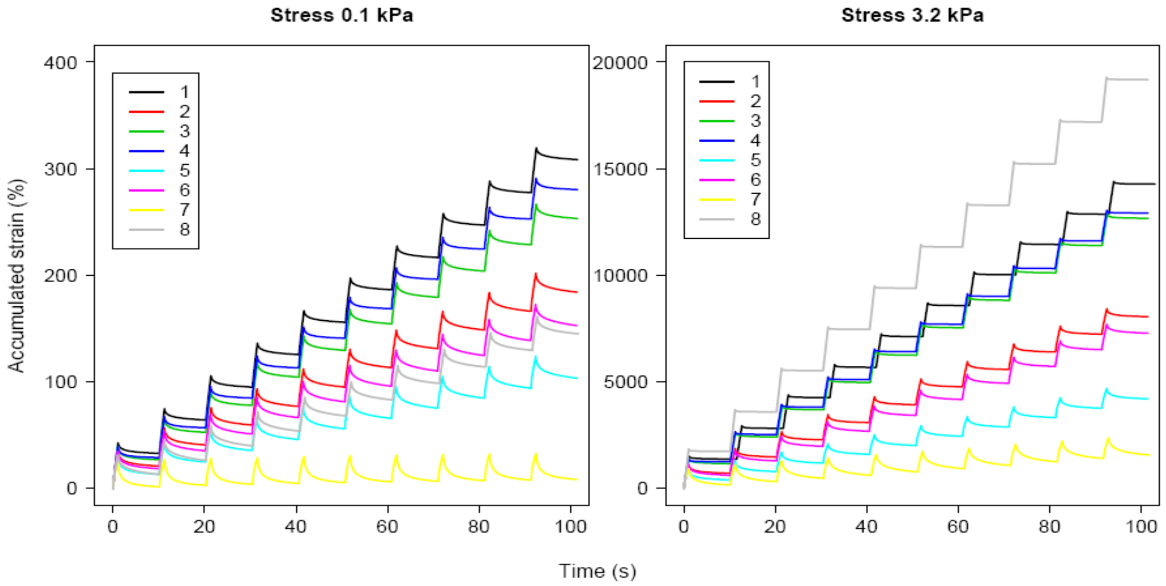

Figure 5: Multiple Stress Creep and Recovery test, modified bitumens after RTFO at temperature 76 °C

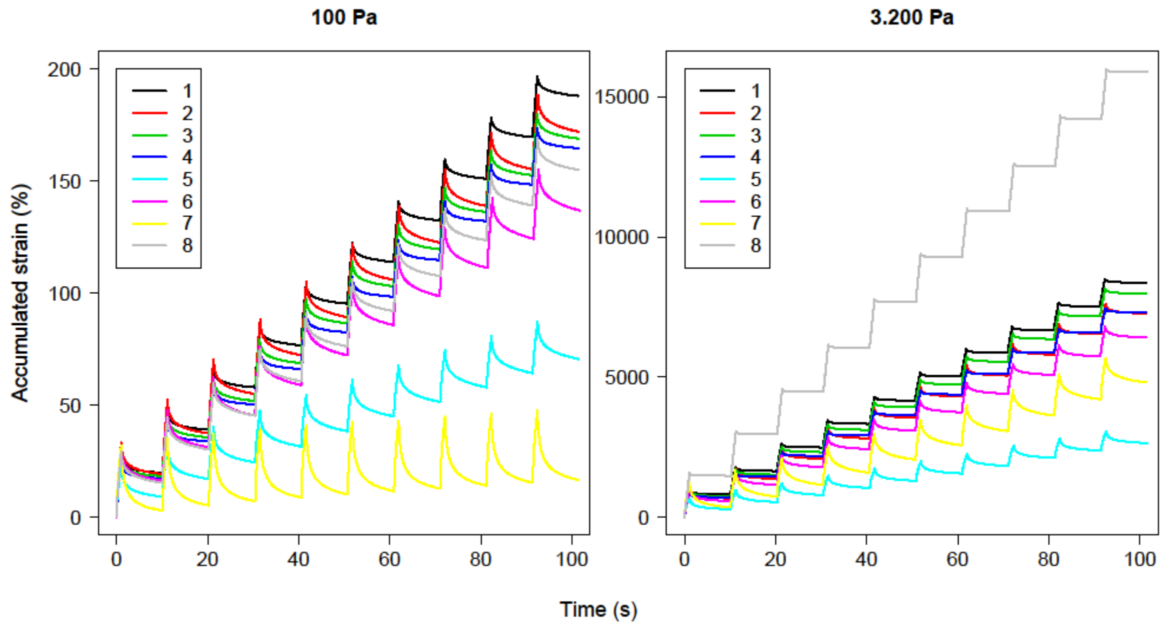

Figure 6: Multiple Stress Creep and Recovery test, modified bitumens after RTFO at critical temperatures

Table 4: Ranking of polymer modified bitumens based on Empirical and Functional Specifications

Sample no.	PMB Grade	Modifier Type	Softening Point	Elastic Recovery	Non Recoverable Creep Compliance			
					PG		Critical temperature	
					unaged	after RTFO	unaged	after RTFO
1	25/55-60	SBS	2-4	2-3	7	N/A	5-7	7
2	45/80-65	RET+SBS	2-4	2-3	2	5-6	4	3-4
3	25/55-60	RET	7-8	7	6	7	5-7	5-6
4	25/55-60	RET+SBS	6	8	3-5	5-6	5-7	5-6

5	45/80-60	RET+SBS	2-4	4-5	1	1	1	2
6	45/80-60	RET	7-8	4-5	3-5	4	3	3-4
7	65/105-75	SBS	1	1	3-5	3	2	1
8	30/70-60	CRmB	5	6	N/A	2	8	8

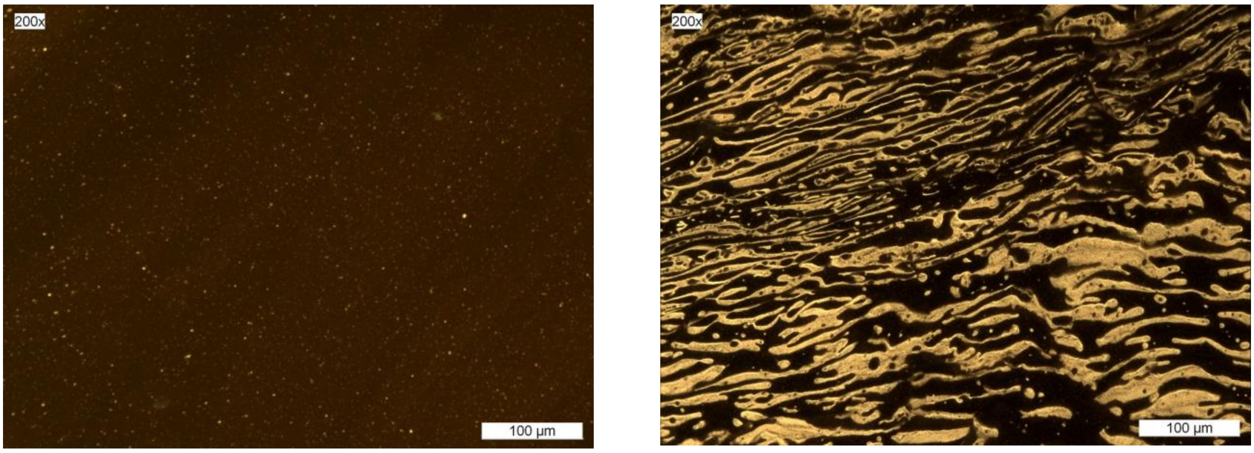

Figure 7: Microstructure of PMB's: PMB 45/80-60 SBS+RET (left) and PMB 65/105-75 SBS (right) after RTFO

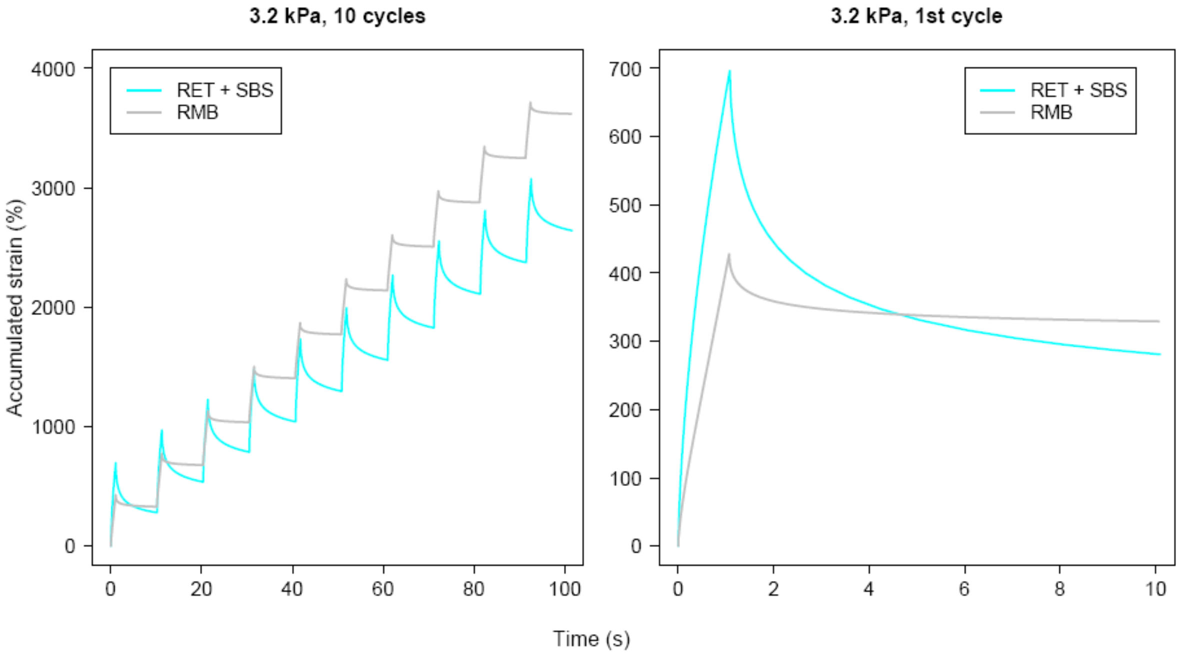

Figure 8: The best ranking of PMB's at temperature 76 °C, after RTFO

4. CONCLUSIONS

Highly modified bitumens needn´t perform as significantly better as other PMB's, in terms of performance tests. The vast difference in softening point and elastic recovery between SBS type PMB 65/105-75 and the blend RET+SBS, type PMB 45/80-60 was narrowed when multiple stress creep is applied and non recoverable creep compliance (Jnr) calculated. As portrayed in **figure 7,** two continuous phases observed may be one of the reasons why is highly modified material failing in terms of repeated loading. The RMB 30/70-60 using crumb rubber in bitumen showed only small changes in critical temperatures, but improved non recoverable creep compliance (Jnr). Rubber modified bitumen after RTFO also showed increase in elastic values due to the continuous reaction or dissolution between particles and binder during the ageing test, but low recovery after the cycle, **figure 8**. Despite the high PG of Crumb Rubber modified bitumen and good non-recoverable compliance the percent recovery is rather weak compare to combination of RET + SBS. It seems that RMB stiffens the material only, rather than contributing to the elasticity of

the material. In terms of MSCR test higher stress shall be applied to reveal the differences.
Decrease of stiffness is recorded at SBS polymer groups, were samples reaching PG 76 for original bitumen, but failing the same grade after RTFO test. The difference between critical temperatures before and after ageing was greater than any other polymers / blends evaluated. It can be explained by changes in colloidal or chemical structure of SBS phase. In this case, SBS polymer groups partially dissolved and reacted with asphaltenes and elasticity remains. Over all, SBS types meet PG 70 requirements after ageing only.
Repeated creep test revealed differences among different materials with similar softening point and initial PG 76. Such variations can not be captured by current empirical European standards. Testing the binders at different stress and application of binder ageing levels dramatically changed the ranking of tested bitumens. It is obvious, that MSCRT or similar methodology using repeated loadings plays important role in terms of bonding and networking during the modification in bitumen and gives better material picture in terms of ageing stability and performance e.g. evaluation of performance at asphalt layers at critical conditions. The response of the binders clearly indicates that some modified binders are more sensitive to stress, number of repetitions and ageing. Loadings at low stress of 100 Pa did not show as important differences as loadings at 3200 Pa. The suggestion is to use even higher stress levels than proposed in prEN 16659. Obtained results shows, that MSCR test has better control in terms of stress and strain on materials fundamental properties and can have a direct relation to the pavement performance than any other empirical techniques.

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