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Rheological properties of SEBS, EVA and EBA polymer modified bitumens

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ABSTRACT

The rheological properties of various polymer modified bitumens were studied. Three bitumens from two different sources were mixed with styrene-ethylene-butylene-styrene (SEBS), ethylene vinyl acetate (EVA) and ethylene butyl acrylate (EBA) copolymers at different polymer contents. The rheological properties of the modified binders were investigated by means of dynamic mechanical analysis and creep test (bending beam rheometer). The results indicated that polymer modification increased binder elastic responses and dynamic moduli at intermediate and high temperatures, and reduced binder complex and stiffness moduli at low temperatures. Polymer modification also reduced temperature susceptibility, glass transition temperature as well as limiting stiffness temperature. The degree of the improvement generally increased with polymer content, but varied with bitumen source/grade and polymer type.

RÉSUMÉ

Les propriétés rhéologiques de divers bitumes modifiés ont été étudiées. Trois bitumes de deux origines différentes ont été mélangés avec les copolymères styrène-éthylène-butylène-styrène (SEBS), éthylène-vinyl-acétate (EVA) et éthylène-butyl-acrylate (EBA), en utilisant différents pourcentages de polymère. Les propriétés rhéologiques des différents liants modifiés ont été examinées au moyen d'essais dynamiques et d'essais de fluage (bending beam rheometer). Les résultats montrent que l'ajout de polymère augmente la réponse élastique du liant et le module dynamique aux températures moyennes et hautes, et diminue le module complexe et le module de rigidité à basse température. L'ajout de polymère diminue la susceptibilité thermique, la température de transition vitreuse et la température de « rigidité limite ». Le degré d'amélioration augmente généralement avec le pourcentage de polymère, mais il varie avec l'origine et la nature du bitume, et le type de polymère.

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1. INTRODUCTION

Rheology is the science which studies the deformation and flow of materials, whether in liquid, melted, or solid form, in terms of the materials' elasticity and viscosity [1]. Bitumen is a viscoelastic material and may exhibit either elastic or viscous behaviour, or a combination of these, depending on temperature and time over which the bitumen is observed. At sufficiently low temperatures and/or high rates of strain (frequencies), bitumen behaves essentially as an elastic solid. As temperature increases and/or loading rate reduces, the viscous property of bitumen becomes more obvious. At sufficiently high temperatures and/or long loading time, bitumen is essentially a *Newtonian* liquid, and can be described by a shear rate independent viscosity value. The rheological properties of bituminous binders may significantly influence the performance of asphalt mixture during mixing, compaction and in service. For this reason, various types of rheological testing are often included in binder specifications.

The rheological properties of bitumens are determined by molecular interactions (molecular forces), which in turn depend on chemical compositions [2-6]. In principle, desired rheological properties of bitumen may be achieved by changing chemical composition using an additive or chemical reaction modification [7]. The addition of polymers is a versatile way of modifying the rheology of bitumens [8-11]. Many types of polymers are available; however, there is no universal polymer and selection should be made according to the specific needs.

This paper presents a rheological investigation of various polymer modified bitumens by using dynamic mechanical analysis and creep test (bending beam rheometer). The effects of base bitumen and polymer type and content on rheological properties are studied.

Editorial Note
Prof. Ulf Isacsson is a RILEM Senior Member, he works at the Royal Institute of Technology, a RILEM Titular Member.

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2. MATERIALS AND TEST PROCEDURES

2.1 Materials

Three bitumens (one B85 and two B180) from two sources (Venezuela and Mexico) were used in this study. They are denoted A, B and C, respectively. The physical properties and chemical characteristics of the base bitumens have been reported elsewhere [12].

The polymers investigated are styrene-ethylene-butylene-styrene (SEBS), ethylene vinyl acetate (EVA) and ethylene butyl acrylate (EBA) copolymers, which are representative of synthetic elastomers (SEBS) and thermoplastics (EVA, EBA). The SEBS polymer contains 29% by mass styrene. The two EVA copolymers used are coded as EVA1 and EVA2. Melt indices (MI) of EVA1 and EVA2 are 6 and 158, and their vinyl acetate (VA) contents are 28 and 18% by mass, respectively.

The polymer modified bitumens were prepared using a low shear mixer at 180°C and a speed of 125 rpm. The mixing time was two hours. The process for preparing the modified binders exposed the bitumen to high temperature and air for an extended time, which led to hardening of the bitumen. For the accurate evaluation of polymer effects, the base bitumens were also subjected to the same treatment as the polymer-bitumen blends.

2.2 Test methods

Dynamic mechanical analysis (DMA): DMA with frequency sweeps (from 0.1 to 100 rad/s) at 25 and 60°C and temperature sweeps (from -30 to 135°C) at 1 rad/s were performed using a rheometer. Parallel plates, diameter 8 and 25 mm, were used in a temperature range of -30 to 50°C and 40 to 135°C, respectively. The test procedure has been described previously [13].

Low-temperature creep test: Creep tests were carried out at four different temperatures (-35, -25, -15 and -10°C) using a bending beam rheometer. The rheometer was developed in the SHRP (*Strategic Highway Research Program*) [14]. In tests, the binder was heated to fluid condition, poured into the mold and then allowed to cool at room temperature for about 90 minutes. The sample was then cooled to approximately -5°C for 1 minute and demolded. After demolding, the sample beam (125 mm long, 12.5 mm wide and 6.25 mm thick) was submerged in a constant-temperature bath and kept at each test temperature (starting at -35°C) for 30 minutes. A constant load of 100 g was then applied to the rectangular beam of the binder, which was supported at both ends by stainless steel half-rounds (102 mm apart), and the deflection of center point was measured continuously. Creep stiffness (S) and creep rate (m) of the binders were determined at several loading times ranging from 8 to 240 seconds.

3. RESULTS AND DISCUSSION

3.1 Viscoelastic properties

In DMA, a sinusoidally varying shear strain is imposed on the sample and the amplitude of the resulting shear stress is determined. The ratio of the peak stress to the peak strain is defined as the complex modulus (G^*), which is a measure of the overall resistance to deformation of a material. The in-phase and out-of-phase components of G^* are defined as the storage modulus (G') and the loss modulus (G''), respectively. G' is proportional to the stress in-phase with the strain and provides information on the elastic responses of a material, while G'' is proportional to the stress out-of-phase with the strain and is associated with viscous effects. The phase difference between the stress and strain in an oscillatory deformation is defined as phase angle (δ). This parameter is a measure of the viscoelastic character of the material. A purely viscous liquid and an ideal elastic solid demonstrate δ of 90° and 0°, respectively. The viscoelastic parameters of bitumens are functions of temperature and frequency, which may be modified by the addition of polymers.

In Figs. 1 and 2, the temperature and frequency dependences of G' and δ are illustrated. As can be seen, polymer modification increases bitumen elastic responses at intermediate (0 - 40°C) and high temperatures ($\geq$ 40°C), as evidenced by increased G' and decreased δ. The temperature susceptibility (Fig. 1) and frequency susceptibility (Fig. 2) of G' are also reduced by polymer modification.The improvement increases with polymer content, but varies widely with the type of polymers and the base bitumens. For a given polymer content, the increase in the elastic responses is observed to be highest for the modified bitumens with SEBS, while it is the lowest for those with EVA, as indicated in Figs. 1 and 2, as well as in Table 1. The increased elastic responses are mainly due to polymer networks (cross-links and entanglements) and/or polymer crystallinity. When sufficient polymer networks are formed in the modified binders, *e.g.* in a continuous polymer phase, four regions, glassy, transition, plateau, and terminal or flow, are observed in G', and correspondingly, a maximum and a minimum are observed in δ (Fig. 1).

The four regions can be associated qualitatively with different kinds of molecular responses. In the glassy region, the binder possesses the highest modulus and is hard and brittle, with little or no intermolecular movement possible. In the transition region, the binder is resilient, and there is sufficient thermal energy to allow for localized molecular rotation and slipping. In this region, the modulus falls off rapidly as secondary intermolecular forces are overcome. In the plateau region, the modulus changes little with temperature, and there are still significant molecular entanglements, while local sections are very mobile. In the flow region, molecular interactions have less energy than the thermal energy applied and significant slippage occurs.

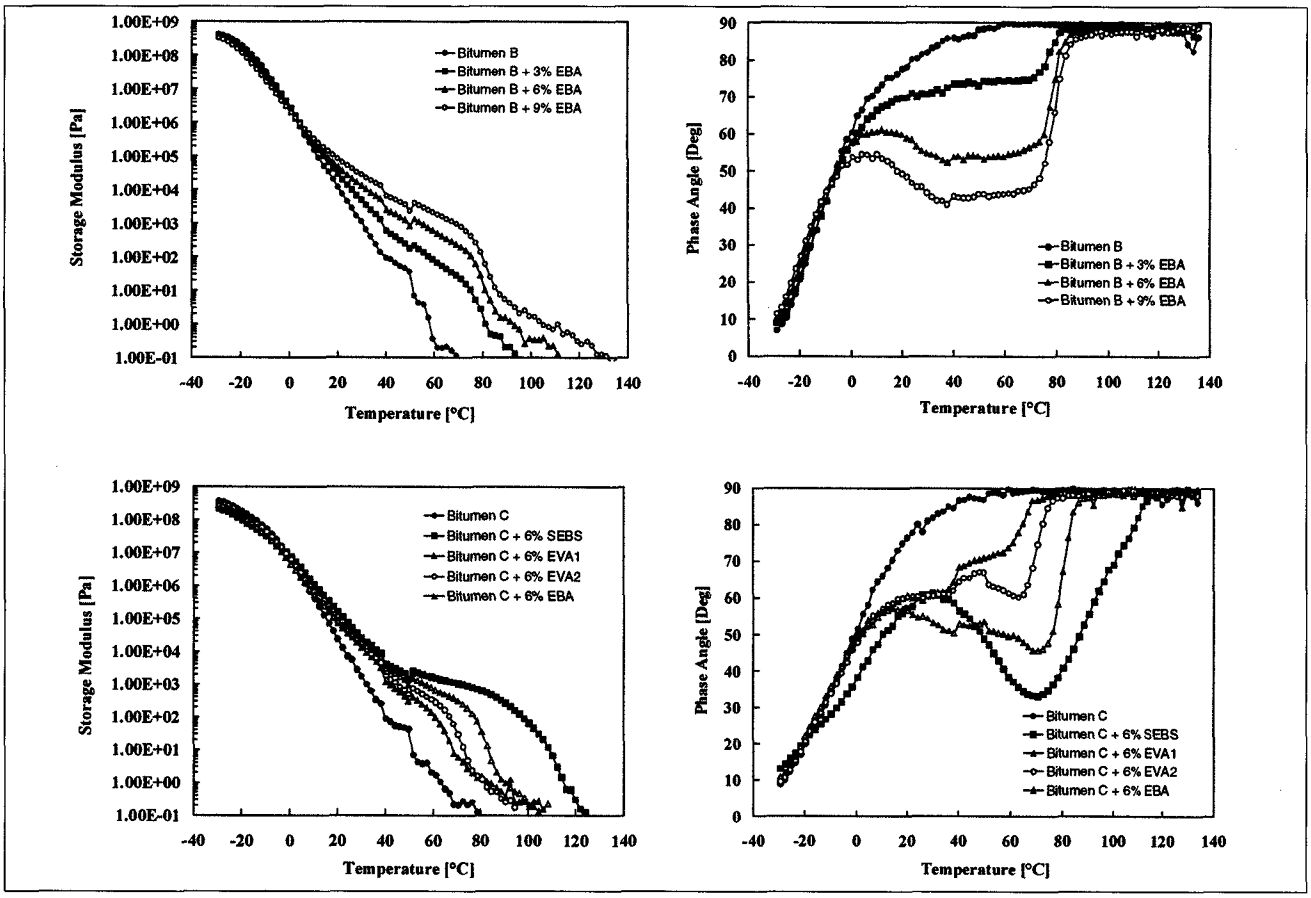

Fig. 1 – Storage modulus and phase angle at 1 rad/s as a function of temperature.

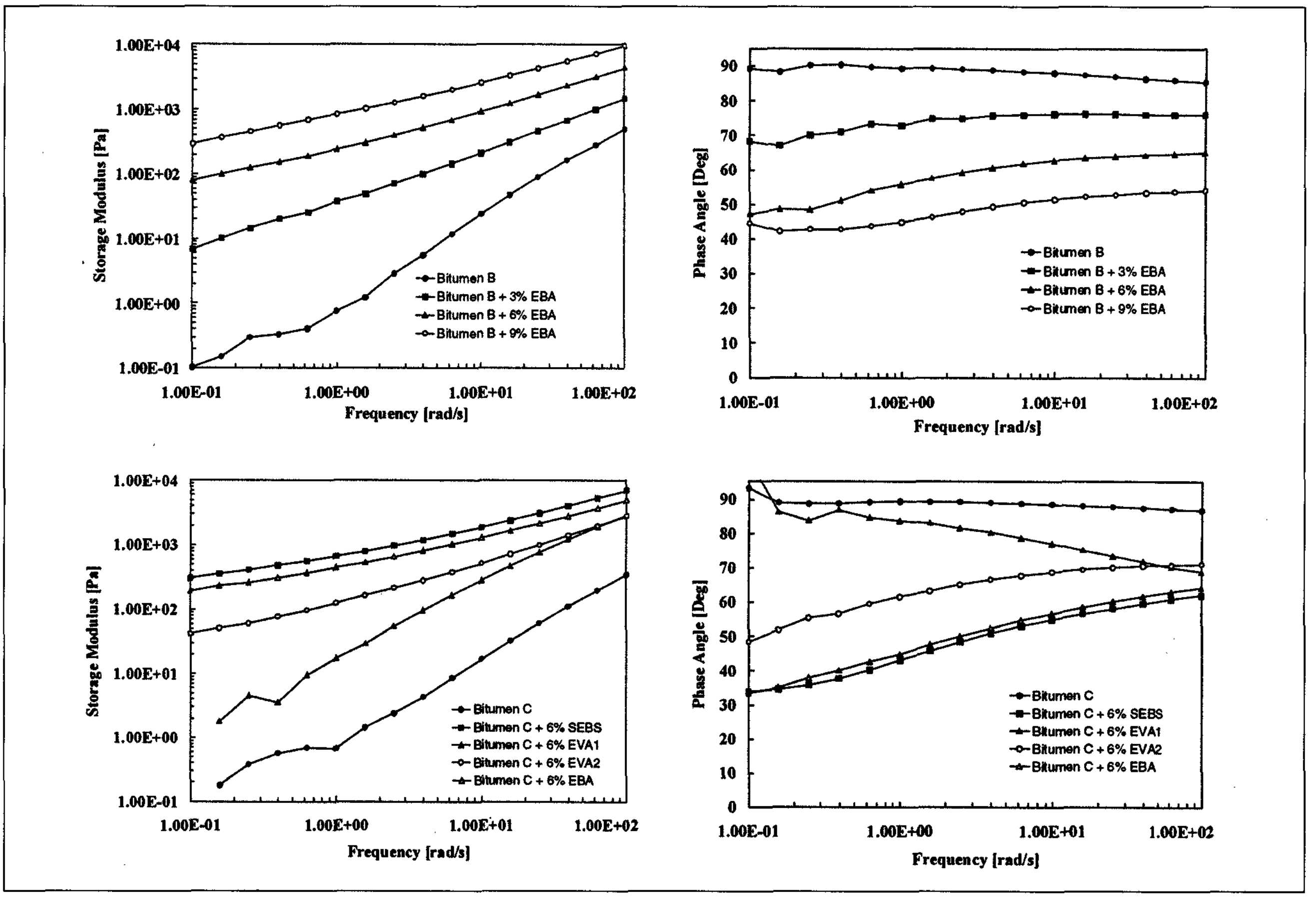

Fig. 2 – Storage modulus and phase angle at 60°C as a function of frequency.

Table 1 – Storage modulus and phase angle at 60°C and different frequencies

Binder	Storage Modulus G′ @ 60°C, Pa			Phase angle δ @ 60°C, Deg		
	1 rad/s	10 rad/s	100 rad/s	1 rad/s	10 rad/s	100 rad/s
Bitumen A	4	128	1970	88.8	86.2	83.1
Bitumen A + 6% SEBS	1420	5420	19100	53.2	52.6	57.1
Bitumen A + 6% EVA1	46	833	6640	82.8	72.3	63.5
Bitumen A + 6% EVA2	115	666	4400	70.1	71.5	70.0
Bitumen A + 6% EBA	628	2340	1050	53.9	60.3	62.5
Bitumen B	1	24	488	89.4	88.0	85.5
Bitumen B + 3% SEBS	5	137	2290	87.9	83.4	75.1
Bitumen B + 6% SEBS	454	1780	8370	55.2	60.9	61.4
Bitumen B + 9% SEBS	2090	5840	19700	41.6	48.2	51.4
Bitumen B + 3% EVA1	4	71	993	86.6	83.7	78.8
Bitumen B + 6% EVA1	13	305	3020	85.2	76.1	66.7
Bitumen B + 9% EVA1	73	952	6900	79.2	69.2	58.9
Bitumen B + 3% EVA2	8	64	758	83.4	83.9	81.3
Bitumen B + 6% EVA2	14	164	1680	82.6	79.3	74.5
Bitumen B + 9% EVA2	37	375	3160	79.4	75.1	69.7
Bitumen B + 3% EBA	37	209	1470	72.9	76.2	76.1
Bitumen B + 6% EBA	240	916	4360	55.9	62.8	65.1
Bitumen B + 9% EBA	841	2570	9430	44.9	51.6	54.2
Bitumen C	1	17	344	89.4	88.5	86.7
Bitumen C + 6% SEBS	673	1900	7060	43.1	55.0	62.1
Bitumen C + 6% EVA1	18	286	2910	83.4	77.1	68.9
Bitumen C + 6% EVA2	126	526	2830	61.6	68.8	71.2
Bitumen C + 6% EBA	447	1290	4940	44.9	56.8	64.2

Different degrees of prominence may be observed in the four regions, depending on the chemical and physical nature (*e.g.* molecular weight, microstructure and network density) of the binders. The density of polymer networks is associated with several factors such as the chemical compositions of the bitumen and the characteristics and content of the polymer. It is also influenced by the temperature. For EVA and EBA modified binders, the crystalline portions (packed polyethylene segments) of the polymers may melt and the polymer networks weaken at temperatures higher than about 60°C (EVA) or 80°C (EBA), leading to a pronounced decrease in G′ and a sharp increase in δ (Fig. 1). In the case of SEBS modified binders, the dramatic changes in G′ (decrease) and δ (increase) are observed at temperatures higher than about 100°C, where the physical cross-links of polystyrene diminish rapidly.

Polymer modification of bitumen rheology is also identified in Fig. 3, where BLACK diagrams (G* as a function of δ) are illustrated. These diagrams are gener-

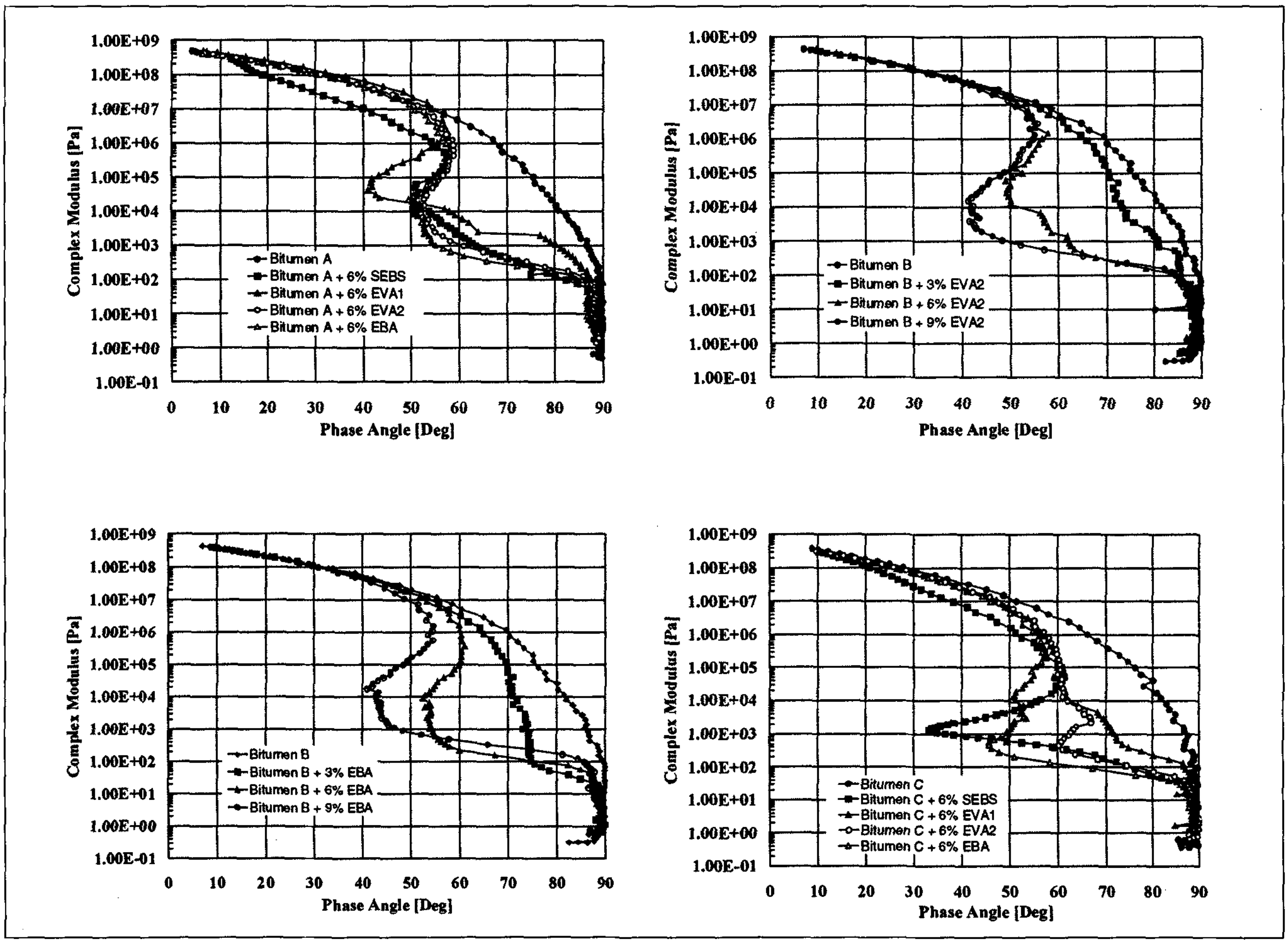

Fig. 3 – Complex modulus as a function of phase angle.

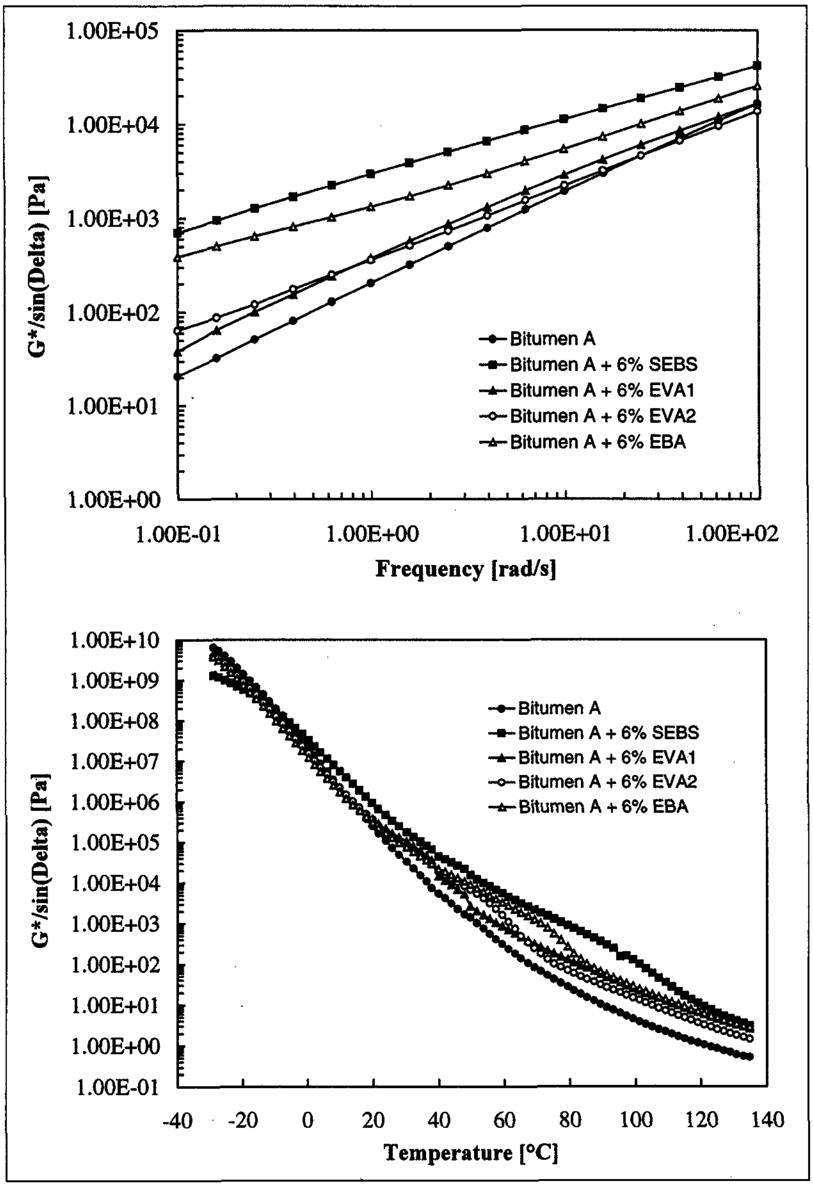

Fig. 4 – G*/sinδ as a function of frequency (at 60°C) and temperature (at 1 rad/s).

ated with temperature sweeps (from -30 to 135°C) at 1 rad/s. As can be seen, for the base bitumens and modified binders containing 3% by mass of polymer, G* decreases smoothly as the increasing temperature changes the behaviour of the binder from essentially solid elastic (δ → 0°) to completely liquid viscous (δ = 90°). A difference between the base bitumens and the modified binders with 3% by mass of polymer is observed at δ higher than about 60°, where G* is mainly contributed by its viscous part G". This indicates that, at a low polymer content (3% by mass in this study), the nature of the polymers shows only a secondary effect on the rheological properties of the modified binders, and the behaviour of the modified binders remains close to that of the base bitumens. However, after modification with a sufficiently high polymer content (≥ 6% by mass), the binders change fundamentally in their rheological behaviour. As indicated in Fig. 3, for all the modified binders containing 6 or 9% by mass of polymer, a substantial decrease in δ (a substantial increase in the elastic response) is observed as G* decreases with increasing temperature. In these cases, the rheological response of the modified binders is mainly imposed by the viscoelastic properties of the polymers, those of the bitumens waning considerably.

These observations imply that the rheological properties of polymer modified binders are probably governed by their continuous phase. Fluorescence photomicrographs indicated that the modified binders with a low polymer content displayed a continuous bitumen phase, while those containing a high polymer content (≥ 6% by mass) showed a continuous polymer phase [15]. The rheological changes are also influenced by characteristics of the bitumens and the polymers. For the modified binders with SEBS, G* deviates from that of the base bitumens at δ as low as 10° (corresponding to binder glassy state at low temperatures). However, the curves of the modified binders with EVA and EBA are almost the same as those of the base bitumens, even at the point where δ = 50° and G* = 10 MPa. These differences suggest that SEBS may improve bitumen rheology over a wide temperature range, while EVA and EBA show their effect mainly at high temperatures.

Due to the increased elastic component of the modified binders, the SHRP rutting parameter, G*/sinδ [16], is improved. However, care should be taken in using this parameter for evaluating the contribution of a modified binder to rutting performance. The reason is that G*/sinδ of a polymer modified binder is more dependent on testing conditions than a conventional bitumen, and for different modified binders, the plots of G*/sinδ as a function of frequency or temperature may cross. This phenomenon is shown in Fig. 4.

By extrapolating the value of G* for δ = 0 in Fig. 3, the glassy modulus G_g* of the binders may be obtained. As can be seen, the values of G_g* are close to 1 GPa and are insignificantly influenced by polymer modification. The 1 GPa reflects the rigidity of the carbon hydrogen bonds as the bitumens reach their minimum thermodynamic equilibrium volume [17]. However, the influence of polymer modification on binder low-temperature properties may be demonstrated by DMA glass transition temperature (Tg), which is defined as the temperature at peak G". As shown in Table 2, the addition of a high content (≥ 6% by mass) SEBS results in a significant reduction (up to 14°C) in Tg of the binders. In contrast, relatively small changes (a few °C) in Tg are caused by EVA and EBA modification. Table 2 also indicates that, at Tg, the rheological parameters (*e.g.* G* and δ) vary considerably. However, the differences are smaller compared with those at high temperatures. This is probably because, in the glassy state, only very short segments of the molecules are invloved in molecular motion, while the motion of large segments is frozen-in, leading to inhibition of polymer effects.

Table 2 – Glass transition temperature and corresponding dynamic moduli and phase angle obtained using DMA at 1 rad/s

Binder	T_g from Peak G″, °C	G″ peak value, MPa	G′ at T_g, MPa	G* at T_g, MPa	δ at T_g, Deg
Bitumen A	-16.3	70.6	205	217	19.0
Bitumen A + 6% SEBS	-30.6	72.0	321	329	12.6
Bitumen A + 6% EVA1	-19.6	85.8	247	262	19.2
Bitumen A + 6% EVA2	-19.7	74.8	253	264	16.4
Bitumen A + 6% EBA	-19.6	71.0	219	230	18.0
Bitumen B	-22.1	75.8	252	264	16.7
Bitumen B + 3% SEBS	-20.5	58.6	179	208	16.0
Bitumen B + 6% SEBS	-32.7	84.8	311	323	15.2
Bitumen B + 9% SEBS	-36.6	86.1	361	371	13.4
Bitumen B + 3% EVA1	-21.7	73.2	224	235	18.1
Bitumen B + 6% EVA1	-23.6	75.6	251	262	16.8
Bitumen B + 9% EVA1	-23.5	74.0	224	236	18.3
Bitumen B + 3% EVA2	-21.5	72.9	231	242	17.6
Bitumen B + 6% EVA2	-23.6	81.0	270	282	16.7
Bitumen B + 9% EVA2	-24.5	73.1	256	267	16.5
Bitumen B + 3% EBA	-21.6	73.9	227	238	18.1
Bitumen B + 6% EBA	-23.5	75.7	245	256	17.2
Bitumen B + 9% EBA	-25.5	72.2	252	262	16.0
Bitumen C	-24.0	67.0	255	264	14.7
Bitumen C + 6% SEBS	-30.7	60.0	286	292	11.9
Bitumen C + 6% EVA1	-23.6	55.5	199	207	15.6
Bitumen C + 6% EVA2	-23.6	61.3	223	231	15.4
Bitumen C + 6% EBA	-25.4	57.5	229	236	14.1

3.2 Low-temperature creep responses

For a flexible pavement, one of the failure modes is low-temperature cracking. Low-temperature cracking is caused by thermal and/or load-induced tensile stresses when these exceed the tensile strength of the pavement material. To reduce the risk of cracking, the binder should have a low stiffness and a high ability of stress relaxation at the lowest pavement temperature [17]. In the SHRP binder specification, a limited creep stiffness (S) and logarithmic creep rate (m-value, which is related to binder stress relaxation ability) have been used as performance-based criteria at low temperatures [18]. The measurements of low-temperature creep responses are conducted at temperatures 10°C above the minimum pavement design temperature.

In this study, the SHRP bending beam rheometer was employed to determine binder low-temperature creep responses at six different loading times (8, 15, 30, 60, 120 and 240 sec) and four different temperatures (-35, -25, -15 and -10°C). Typical examples of creep response are shown in Fig. 5, in which the influence of polymer modification is clearly illustrated. In Table 3, the creep stiffness and m-value obtained at a loading time of 60 sec and four different temperatures are presented. As can be seen, the polymer modified binders generally display a lower creep stiffness than the corresponding base bitumen, especially at temperatures lower than -15°C. The improvement increases with polymer content, but varies with the base bitumen and polymer type. At -35°C and for a given polymer content, the modified binders containing SEBS show a higher reduction in creep stiffness than those with EVA and EBA. However, at the other three low temperatures, varying changes are observed in creep stiffness. Some modified binders (*e.g.* Bitumens A and B modified with SEBS) even show an increased creep stiffness at -10 and -15°C. Moreover, the use of a softer bitumen seems favourable for obtaining higher improvement in creep stiffness (cf. B85 and B180). As regards effect of the polymer modification on binder stress relaxation ability, both increased and reduced m-values are observed, depending on the testing temperature and loading time.

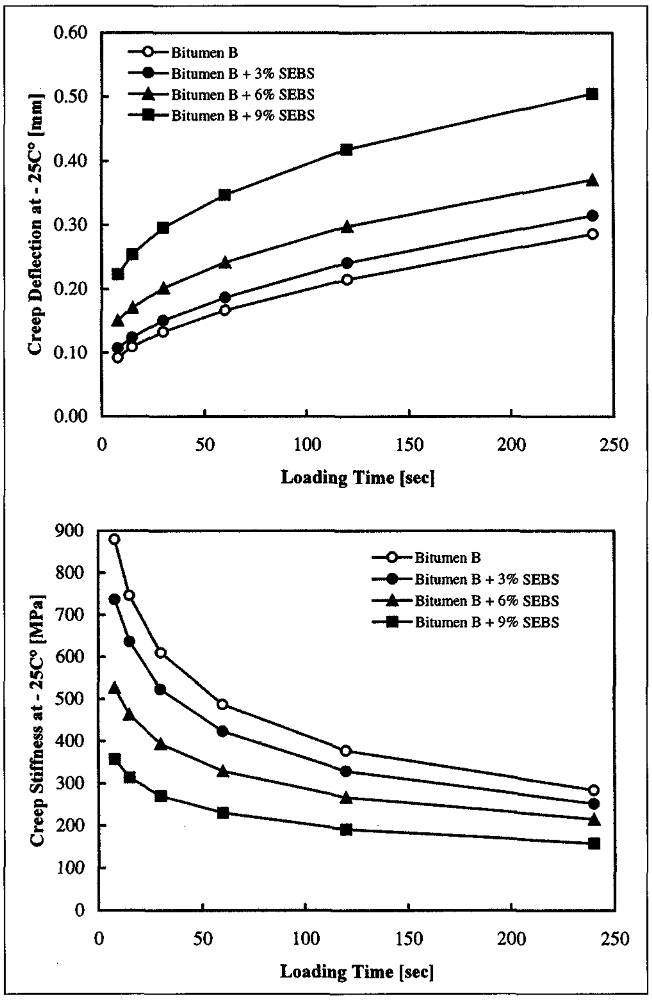

Fig. 5 – Creep response of the base and SEBS modified bitumen B at -25°C.

Table 3 – Creep stiffness and m-value of the binders at a loading time of 60 s and different temperatures

Binder	Creep Stiffness [MPa]				*m*-value			
	-10°C	-15°C	-25°C	-35°C	-10°C	-15°C	-25°C	-35°C
Bitumen A	46	185	789	1590	0.58	0.45	0.26	0.09
Bitumen A + 6% SEBS	80	195	684	1140	0.45	0.38	0.20	0.11
Bitumen A + 6% EVA1	46	132	624	1560	0.54	0.47	0.30	0.13
Bitumen A + 6% EVA2	72	180	790	2000	0.47	0.42	0.25	0.12
Bitumen A + 6% EBA	66	161	656	1620	0.45	0.41	0.26	0.13
Bitumen B	12	54	487	1560	1.00	0.61	0.35	0.15
Bitumen B + 3% SEBS	17	63	423	1180	0.72	0.54	0.33	0.15
Bitumen B + 6% SEBS	27	80	329	774	0.52	0.41	0.28	0.19
Bitumen B + 9% SEBS	27	60	230	575	0.45	0.38	0.25	0.21
Bitumen B + 3% EVA1	12	40	350	1360	0.97	0.64	0.41	0.17
Bitumen B + 6% EVA1	12	36	326	1200	0.77	0.61	0.42	0.19
Bitumen B + 9% EVA1	13	31	302	1210	0.67	0.58	0.46	0.20
Bitumen B + 3% EVA2	13	50	371	1190	0.86	0.56	0.40	0.16
Bitumen B + 6% EVA2	14	48	366	1240	0.67	0.54	0.38	0.18
Bitumen B + 9% EVA2	10	27	265	1140	0.70	0.59	0.42	0.19
Bitumen B + 3% EBA	13	45	383	1250	0.82	0.58	0.37	0.18
Bitumen B + 6% EBA	13	38	298	1160	0.59	0.52	0.38	0.19
Bitumen B + 9% EBA	10	27	234	1020	0.58	0.54	0.42	0.21
Bitumen C	33	99	437	931	0.59	0.49	0.32	0.19
Bitumen C + 6% SEBS	43	97	333	765	0.52	0.42	0.28	0.18
Bitumen C + 6% EVA1	38	89	357	958	0.51	0.47	0.31	0.19
Bitumen C + 6% EVA2	37	94	411	1040	0.51	0.44	0.32	0.19
Bitumen C + 6% EBA	24	60	167	875	0.50	0.35	0.16	0.20

Consequently, no definite conclusions could be drawn on this matter.

As illustrated in Fig. 6, creep stiffness statistically (risk level 5%) correlates with complex modulus. The linear relationship becomes better as loading conditions are similar for creep test and dynamic mechanical analysis (frequency 1 rad/s corresponds to a loading time of approximate 6.3 s).

To avoid low temperature cracking, the SHRP binder specification [18] requires that creep stiffness must not exceed 300 MPa at a loading time of 60 seconds. A resulting parameter for the evaluation of low temperature properties is the temperature at 300 MPa creep stiffness, or the limiting stiffness temperature. As indicated in Fig. 7, polymer modification reduces the limiting stiffness temperature. The modification is influenced by polymer type/content and bitumen source/grade. This temperature may statistically (risk level 5%) correlate with DMA glass transition temperature (R = 0.50). The low correlation coefficient is due to the four modified binders containing a high content (6 and 9% by mass) of SEBS, which deviate from the others in a significant way. The correlation becomes much better (R = 0.91) if those modified binders are excluded from the statistical analysis, as illustrated in Fig. 8. This indicates that, for some polymers at sufficiently high contents, the rheological properties of the modified binders measured is strongly dependent on the type of test procedure.

4. CONCLUSIONS

The rheological properties of various polymer modified binders have been investigated using dynamic mechanical analysis and creep test (bending beam rheometer). On the basis of the data and interpretations presented in this study, the following conclusions may be drawn.

Polymer modification increases the elastic response and dynamic modulus of bitumens and reduces their temperature susceptibility at intermediate (0 - 40°C) and high (≥ 40°C) temperatures. The degree of the modification generally increases with polymer content. Significant improvement is observed as the content of polymer is sufficiently high (6% by mass in this study). Other factors affecting the rheological behaviour of the binder are the characteristics of the base bitumen and the polymer. For a given base bitumen, the effectiveness of the four polymers tested may be ranked as SEBS,

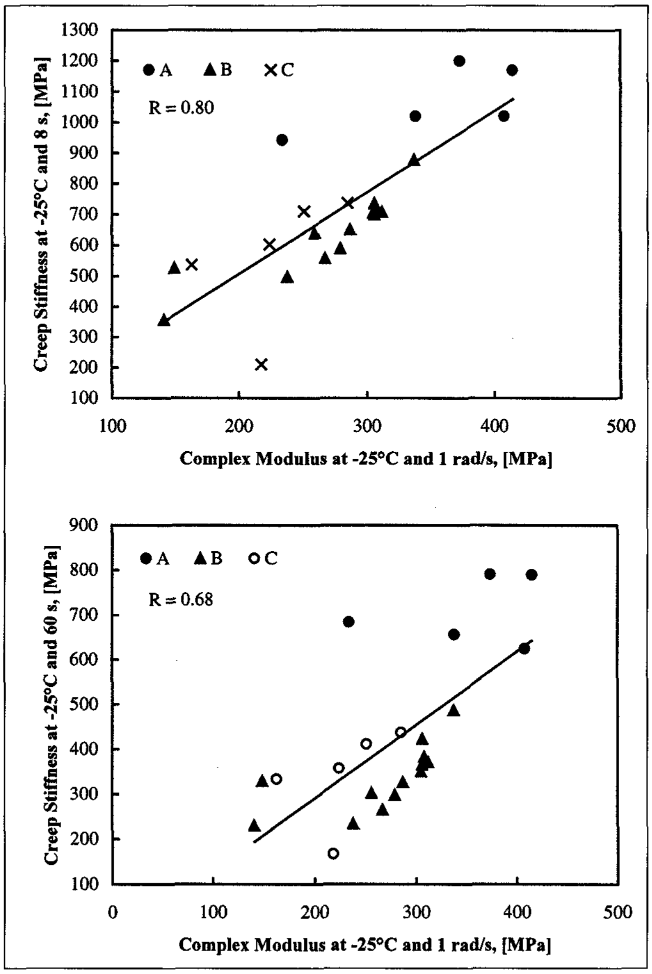

Fig. 6 – Relationship between creep stiffness and complex modulus.

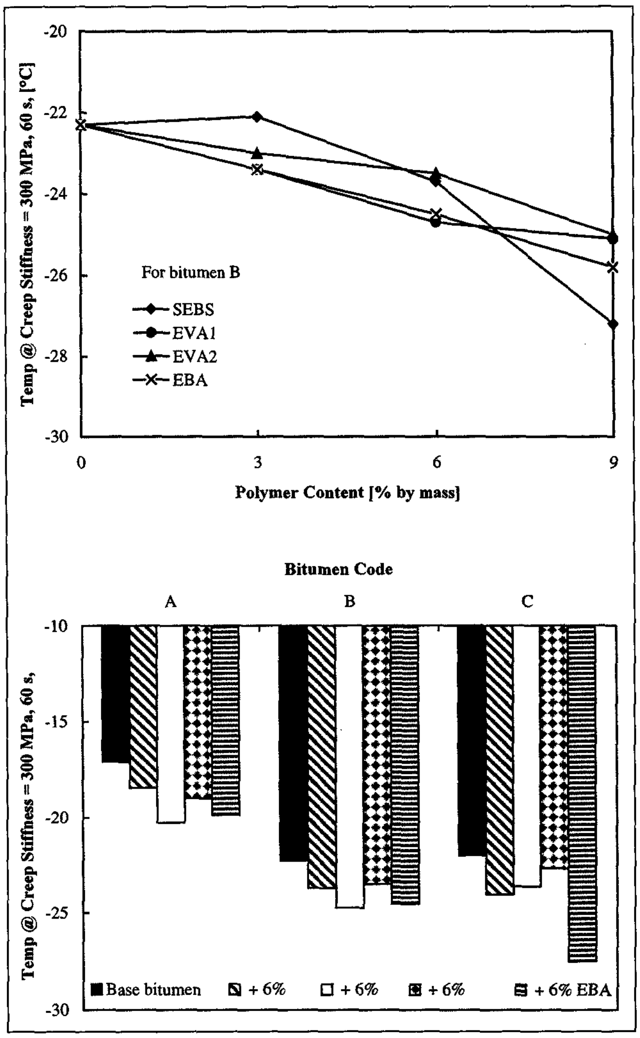

Fig. 7 – Influence of bitumen source/grade and polymer type/content on the temperature at 300 MPa creep stiffness.

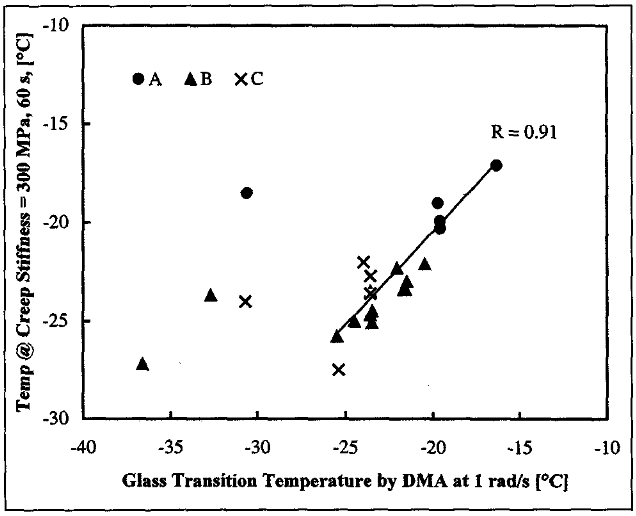

Fig. 8 – Temperature at 300 MPa creep stiffness as a function of DMA glass transition temperature.

EBA, EVA2 and EVA1, SEBS being the most effective. The improved dynamic rheological properties should be favourable for resistance to rutting at high temperatures.

Polymer modification also improves the low-temperature properties of bitumens, as indicated by decreases in creep stiffness, glass transition and limiting stiffness temperatures. The relative improvement varies with the base bitumen, polymer type and polymer content. Of the four polymers studied, the SEBS polymer seems the most effective in improving bitumen low-temperature parameters (except for the limiting stiffness temperature), similar to its greatest influence on bitumen high-temperature rheology. Unlike the situation at high temperatures, insignificant differences in the low-temperature parameters are observed between the EVA and EBA modified binders. For a given polymer, the B180 bitumens may result in a relatively higher reduction in creep stiffness than the B85 bitumen. The influences of polymer modification may also vary with testing conditions (temperature and loading time). The improved low temperature parameters should be favorable with respect to asphalt cracking performance in practice.

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