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Thermal Analysis & Rheology

The Use of the Method of Incomplete Creep to Assess the Resistance to Rutting of Bituminous Materials

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Abstract

One of the most difficult problems in materials testing is the rapid evaluation of long term material performance. An effective test method requires that the material properties used as predictors are both relevant and convenient to determine. For bituminous materials various rheological functions have been proposed as being appropriate for the assessment of resistance to rutting. For example the SHRP standard procedures, recently introduced in the United States, require the use of the norm of the complex modulus divided by the sine of the phase angle, $|G^*| / \sin\delta$. Other workers suggest that the low shear limit of the viscosity η_0, is likely to be more generally applicable. Unfortunately, for some materials, including many polymer modified bitumens, dynamic methods of determining η_0 cannot be used; an alternative is to use a creep test. A drawback of such a test in its normal operating mode is that it needs to be run until steady state has been achieved. In the method of incomplete creep the use of the Boltzmann superposition principle makes this condition unnecessary. In this paper the method is used to determine η_0 for a range of modified and unmodified bitumens. It is further shown how $|G^*| / \sin\delta$ can be calculated numerically from such a test.

1. Introduction

An efficient road surface must be resistant to degradation under both thermal cycling and mechanical impact. It must neither crack nor rut under normal operating conditions, and preferably over a period of greater than ten years [1,2]. Yet it is clearly impractical to measure the performance of a prospective new material for this length of time. Test methods are required which can be carried out rapidly, but which allow the prediction of the long term material performance.

To this end several rheological tests, which are normally performed on the bituminous binder, have been devised for the assessment of the resistance to rutting of asphalts. But which rheological property best predicts the rutting resistance has been the subject of much debate in recent years. The standard procedures recently introduced in the United States by the Strategic Highways Research Program (SHRP) require the use of the norm of the complex modulus divided by the sine of the phase angle [3]. Other workers have suggested the low shear limit of the viscosity [4]. It might be useful to discuss briefly how these properties are determined, and the reasons that they are regarded as being important.

The storage modulus is obtained from an oscillatory test, in which a sinusoidal shear stress, σ, is placed on a standard sized sample. The resulting shear strain, γ, is monitored. The complex modulus, G^*, is defined as the stress amplitude, σ_0, divided by the strain amplitude, γ_0 , and can be decomposed into a norm, $|G^*|$, and phase angle, δ. It should be noted that $|G^*|$ is a function of both the frequency and amplitude of the applied stress. The SHRP procedures overcome this by requiring its measurement within the linear regime, where it is amplitude independent, and at an angular frequency, ω, of 10 rads / sec.

Since $|G^*|$ is a measure of the stiffness of the material, it was considered by the authors of the SHRP procedures to provide a reasonable basis for an assessment of the rutting resistance. The term in the phase angle was included as it was perceived that the smaller the value of δ, i.e. the greater the elasticity, the greater the recovery on removal of the load. The frequency was chosen to represent the inverse of the typical loading duration under traffic.

Most bituminous materials are shear thinning, that is they exhibit viscosities which decrease monotonically with applied stress. However, at low stresses this decrease is in most cases negligible, and the flow properties of the material comply with Newton's law of viscous flow, i.e.:

$$\sigma = \eta_0 \dot{\gamma} \tag{1}$$

where η_0 is the low shear limit of the viscosity and $\dot{\gamma}$ is $d\gamma/dt$. It can be determined by extrapolation from a dynamic (oscillatory) experiment, since it is a requirement of continuum mechanics that $G^*(\omega) / \omega\big|_{\omega \to 0} = \eta_0$. Since several data points acquired at low frequencies may be required for an efficient extrapolation, the experimental procedure may take many minutes to perform; to make matters worse, the SHRP procedure requires data taken over a range of temperatures.

The more usual, and quicker, way to determine η_0 is to perform a creep test, in which a low stress is placed on the sample and the viscosity calculated from the shear rate at steady state. However, in its standard operation, a creep test does not directly provide G* / sin δ. Furthermore, it is not always clear when steady state has been achieved; this difficulty can result in tests of unnecessarily long duration.

It would be of obvious benefit if a simply conducted and rapid method of determining both G* / sin δ and η_0 were available. In this paper it is shown that the use of the Boltzmann superposition principle can circumvent the steady state requirement of a creep test, and that G* / sin δ can be simply calculated from creep data.

2. Theoretical

2.1 The standard creep test

In its standard mode a creep test is straightforward to perform. An instantaneously applied stress is placed on the sample and held constant and the strain response is monitored. Formally, the stress history can be written as:

$$\sigma(t) = \sigma_0 h(t) \tag{2}$$

where h(t) represents the unit (Heaviside) step function, with h = 0 for t < 0, and h = 1 for t > 0 (Figure 1).

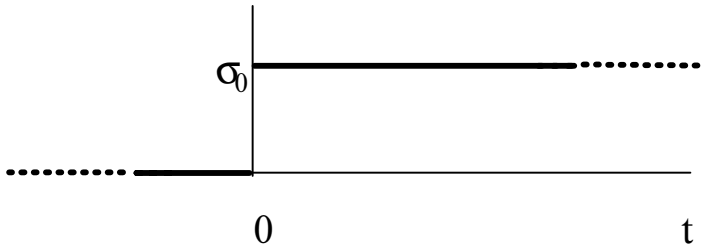

Figure 1: Stress history for a standard creep test

It is usual to quote the results in the form of the creep compliance J(t) = γ / σ, and it is standard practice for the applied stress to be sufficiently low for the compliance to be within the linear regime (stress independent).

J(t) can be considered to be the sum of three separate components [4], i.e.:

$$J(t) = J_0 + J_d(t) + J_v(t) \tag{3}$$

where J_0 is the instantaneous compliance, i.e. J at t = 0, $J_d(t)$ is the delayed compliance and $J_v(t)$ is a viscous contribution. The delayed compliance can often be satisfactorily modelled by a single element Voigt model, defined by the expression:

$$J_d(t) = J^n[1-\exp(-t/\lambda)] \tag{4}$$

where λ is referred to as the retardation time of the element. More usually a number of Voigt elements is required, and:

$$\int_{-\infty}^{\infty} J_d(t) = \Sigma J_n[1 - \exp(-t/\lambda_n)] \tag{5}$$

In the infinite limit this becomes the integral:

$$L(\lambda)[1 - \exp(-t/\lambda)]\, d \ln \lambda \tag{6}$$

where L(λ) is known as the retardation spectrum.
The viscous contribution is given by:

$$J_v(t) = t / \eta_0 \tag{7}$$

It is clear that for any non-zero value of J_d, the exponentials demand that steady state is never strictly achieved. However, pseudo steady state can be defined as the point at which the contribution from J_d falls below the instrument resolution, and it is then a simple matter to decompose J(t). J_0 is immediately accessible by extrapolation. J (t) can be obtained from the steady state response, and therefore η_0 from its inverse gradient (reference [5] provides more complicated procedures which circumvent the difficulties involved in, for example, the extrapolation of a curve). Subtracting these contributions from J(t) gives $J_d(t)$. From Equation (5) it can be seen that $[J_d(\infty) - J_d(t)]$ is the sum of a number of exponential functions. A plot of $\ln [J_d(\infty) - J_d(t)]$ against time will be linear in the long time region, where the response of the Voigt element with the longest retardation time dominates. J and λ for this element can be simply obtained from this part of the curve, and its contribution subtracted. The process can then be repeated for elements with successively decreasing retardation times.

2.2 Numerical calculation of $G^* / \sin\delta$

By definition $G^*(\omega) = 1 / J^*(\omega)$, and $J^*(\omega) \sin\delta = J''(\omega)$, the loss compliance, so $G^* / \sin\delta = 1 / J''$. But reference [6] gives:

$$J''(\omega) = \sum_n \frac{J_n \omega \lambda_n}{1 + \omega^2 \lambda^2} + \frac{1}{\omega \eta_0} \tag{8}$$

At the SHRP specified frequency of 10 rads/sec, $\omega^2\lambda^2 >> 1$ and, with the viscosity of the nth Voigt element, η_n, defined as λ_n / J_n, Equation (8) becomes

$$\frac{G^*(10)}{\sin\delta} = 10\left(\frac{\eta_0 \sum_n \eta_n}{\sum_n \eta_n + \eta_0} \right) \tag{9}$$

Usually the procedure described above provides values for up to four Voigt elements, so $G^* / \sin\delta$ can be easily calculated for most creep curves.

2.3 The method of incomplete creep

The method of incomplete creep has been described in detail by Meissner [7], and will only be summarized here. The Boltzmann superposition principle states that the effects of mechanical history are linearly additive. The strain response of the material to each element in the stress history can therefore be considered independently.

A creep test is commonly performed by maintaining the stress at σ_1 for a finite length of time, then removing it. The stress history in this case can be written as:

$$\sigma(t) = \sigma_0\{h(t) - h(t - t')\} \tag{10}$$

where t' is the time at which the stress is removed, and the unit gate function $\{h(t) - h(t - t')\} = 0$ for $t < 0$, 1 for $t' > t > 0$, and 0 for $t > t'$.

Since zero stress can be regarded as the resultant of two stresses of equal magnitude but opposite direction, so:

$$\sigma(t) = \sigma_1 + \sigma_2 \tag{11}$$

Where σ_1 and σ_2 are equal in magnitude to σ_0, but are of opposite sign to each other, and start at different instants (Figure 2)

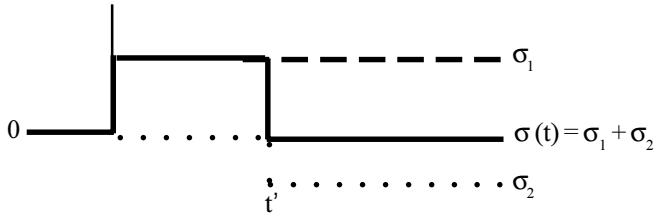

Figure 2: Stress history for creep test with stress removed at time t'.

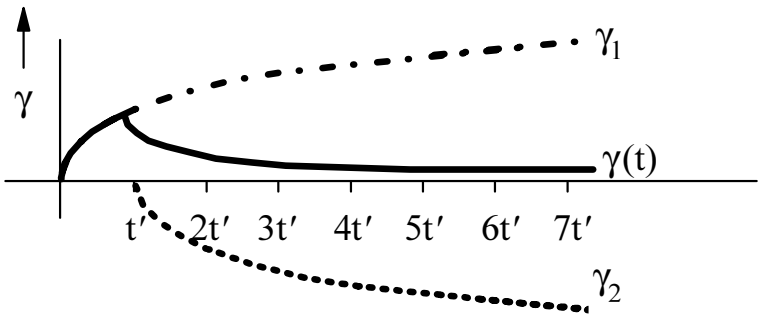

Figure 3: Strain response to incomplete creep test

In accordance with the Boltzmann superposition principle, γ_1, the strain response from σ_2 will be equal to γ_2, the strain response from σ_1, but of course will be of opposite sign, and will start at t′. The total strain, $\gamma = \gamma_1 + \gamma_2$ (Figure 3)

But $\gamma_2(t = t') = -\gamma_1(t = 0) = 0$; $\gamma_2(2t') = -\gamma_1(t')$; $\gamma_2(3t') = -\gamma_1(2t')$ and so on. So $\gamma(xt') = \gamma_1(xt') - \gamma_1((x-1)t')$. Since $\gamma(t)$ is being monitored, and $\gamma_1(t')$ is known, $\gamma_1(2t')$ can be calculated, followed by $\gamma_1(3t')$, and so on. In this manner the complete creep curve can be regained, without the difficulty of ascertaining when steady state has been achieved; it is simply when $\gamma(t)$ becomes horizontal.

3. Experimental details

3.1 Sample details

Two samples were used in this investigation. Sample A was a 70 PEN unmodified bitumen, Sample B was modified by the addition of 10 % SBS block copolymer.

The instrument used was a AR 1000 controlled stress rheometer, supplied by TA Instruments. A 2 cm diameter composite parallel plate geometry was used at 80°C and 60°C with a gap width of 1000μm. At the lower temperatures (40°C to 10°C) a 0.8 cm diameter composite parallel plate was used with a gap of 2000μm. This accords with SHRP recommended practice, which requires the smaller sized plate to obviate artifacts introduced by instrument compliance. It is worth remarking, however, that since the instrument compliance contributes only to J_0, and that this does not occur either explicitly or implicitly in Equation 8, then if the only parameter of interest is J″ (or G* / sin δ), the instrument compliance is immaterial. It is also worth noting that for parallel plate geometries the equations used in the calculation of the rheological parameters are exact within the regime of linear viscoelasticity.

3.2 Experimental procedure

The sample was loaded on the instrument and the temperature raised to 80°C. The platens were brought together using normal force closure, i.e. with the stress imposed by the sample normal to the platens being held below 8 Pa. This is to ensure that stresses induced during closure are not excessive.

Oscillation and creep tests were performed for Sample A at 80°C, 60°C, 40°C, 30°C, 20°C and 10°C. The temperature was raised to 80°C between each temperature change, to remove residual stresses. The sample was allowed to equilibrate at each temperature for five minutes, partly to ensure thermal homogeneity, and partly to allow the decay of any stresses induced during cooling. Oscillation and creep tests on sample B were performed only at 20°C.

For the oscillation tests a stress amplitude of 100 Pa was used throughout; frequency sweeps were conducted from 0.1 to 100 rads / sec. To check that the stress amplitude was within the limit of linearity, each frequency sweep was repeated at an amplitude of 50 Pa, taking a reduced number of data points. In all cases it was observed that the modulus curves for the 50 Pa and 100 Pa amplitudes were coincident.

Following the oscillatory tests, preliminary creep tests were conducted at an applied stress of 50 Pa, which was held for three minutes and was followed by a five minute recovery period. In all cases the curves obtained were coincident with those obtained from the creep tests proper, verifying that the condition of linear viscoelasticity held. For the creep tests proper, a stress of 100 Pa was applied. As the method of incomplete creep was being used, the duration of the creep step, that is the period during which the stress was held, was in theory immaterial. The step was simply allowed to proceed until it was judged that suffient data had been collected to allow the complete curve to be calculated. This varied between samples. The recovery step, during the stress was zero, was then allowed to proceed until the compliance had reached a constant value.

4. Results and discussion

4.1 The physical chemistry of the rheology of bitumen has been discussed in detail elsewhere [8], and only the methodology will be considered here.

Figure 4 shows the loss compliance plotted against angular frequency for both samples at all measured temperatures. The decrease in loss compliance with increasing frequency approximately under a power law is as expected, as is the increase with decreasing temperature. At 10 rads / sec this results in a range of about six decimal orders of magnitude for J″. It is interesting to note that the isothermal comparison showed very little difference between the loss compliances and, therefore, G* / sin δ, of Sample A and Sample B. The SHRP criterion predicts that these binders would perform similarly in their resistance to rutting.

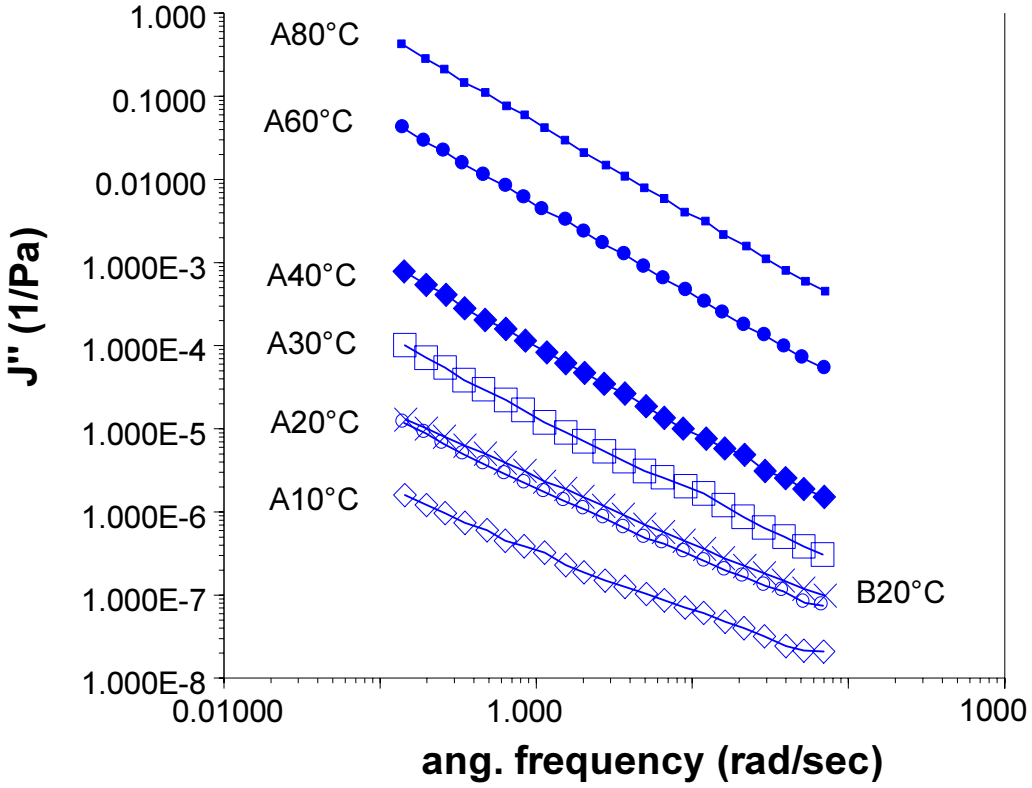

Figure 4: Loss compliance, J″, against angular frequency, ω, for Samples A and B at all temperatures. Crosses indicate Sample B

Figure 5 shows creep and recovery curves for Sample A at 80°C and 60°C. At these temperatures recovery on removal of the stress and, therefore, the contribution from J_d, are negligible. In these circumstances the method of incomplete creep is no more than an exercise in similar triangles, and the curve fitting procedure described above was used only on the primary data sets.

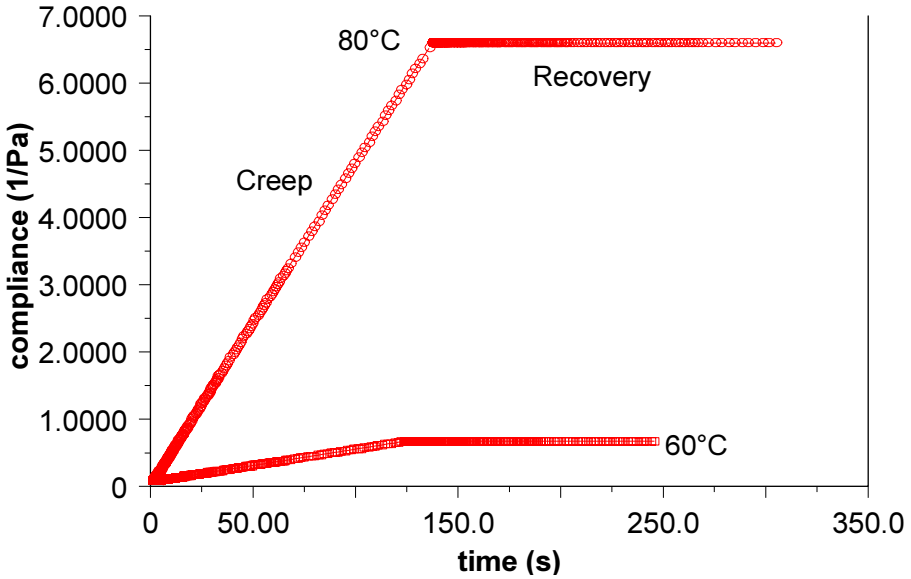

Figure 5: Creep compliance, J(t), against time for Sample A at 80°C and 60°C, showing creep and recovery parts of curve.

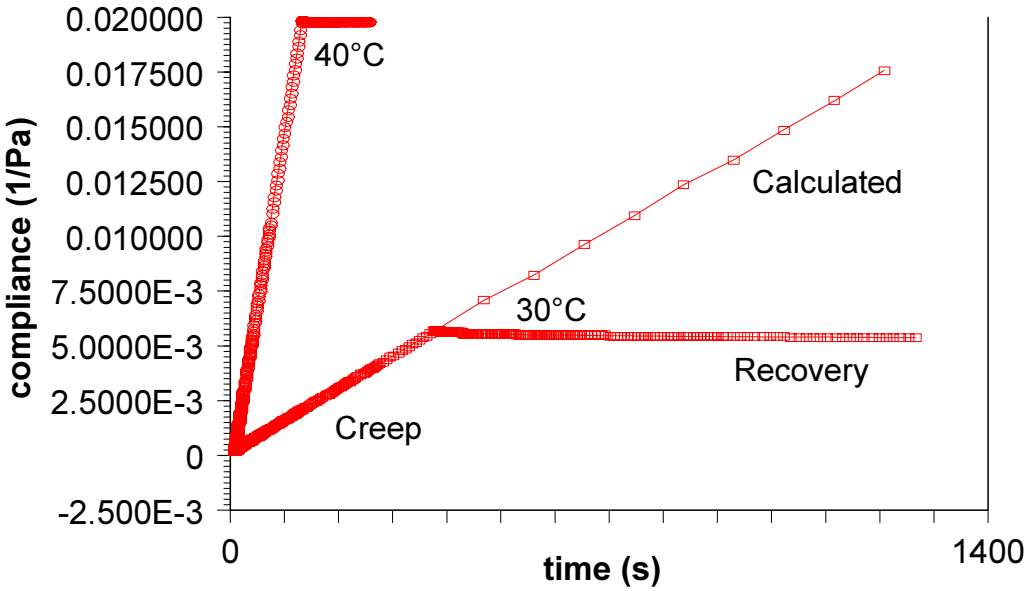

Figure 6: Creep compliance, J(t) against time for Sample A at 40° and 30°C, showing creep, recovery and calculated parts of curve.

Figure 6 shows creep and recovery curves for Sample A at 40°C and 30°C. At 40°C the recovery is still low, and the fitting was on the primary data set. At 30°C significant recovery was observed, and the complete creep curve was calculated using the Boltzmann superposition principle in the manner described above.

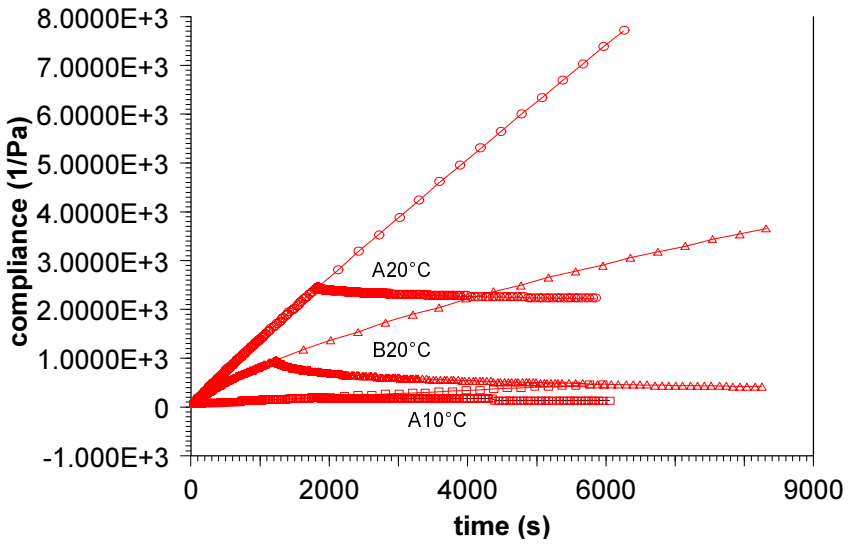

Figure 7: Creep compliance, J(t), against time for Sample A at 20°C and 10°C and Sample B at 20°C, showing creep, recovery and calculated parts of curve.

Figure 7 shows the creep and recovery curves for Samples A and B at 20°C, and Sample A at 10°C. The isothermal comparison between the samples shows that although the measured values of the loss compliance were similar, the viscoelastic properties were markedly different. There is clearly more recovery in the case of sample B, indicating a stronger contribution to the loss compliance from J_d.

Table 1: Measured and calculated values of G*/sin δ for all samples at all temperatures

Sample, Temp. [°C]	η_0 [Pa s]	$\eta_0\omega$ [Pa]	$\Sigma\eta_n$ [Pa s]	J″(ω=10) [1/Pa] calculated	G*(ω=10) /sin δ [Pa] calculated	G*(ω=10) /sin δ [Pa] measured
A, 80	2.09×10	2.09×10^2	negligible	4.78×10^{-3}	2.09×10^2	2.09×10^2
A, 60	2.05×10^2	2.05×10^3	negligible	4.87×10^{-4}	2.05×10^3	1.99×10^3
A, 40	6.28×10^3	6.28×10^4	1.58×10^4	2.23×10^{-5}	4.49×10^4	5.19×10^4
A, 30	6.81×10^4	6.81×10^5	2.19×10^5	1.93×10^{-6}	5.19×10^5	4.72×10^5
A, 20	8.62×10^5	8.62×10^6	1.06×10^6	2.10×10^{-7}	4.75×10^6	3.21×10^6
A, 10	1.58×10^7	1.58×10^8	1.41×10^7	1.34×10^{-8}	7.45×10^7	1.49×10^7
B, 20	3.13×10^6	3.13×10^7	1.50×10^6	6.59×10^{-8}	1.01×10^7	2.29×10^6

The results are summarised in Table 1. Where $\Sigma\eta_n$ is negligible or low (i.e. Sample A at 80°, 60° and 40°), the correspondence between the product of the angular frequency and the zero shear viscosity and G* / sin δ is good (<12% difference).

In the instances of intermediate elasticity (Sample A at 30°C and 20°C) the correspondence with η_0 is poor, but becomes fair (<15% difference) when $\Sigma\eta_n$ is also considered.

Where the elastic contribution to the response is large (Sample A at 10°C, Sample B at 20°C) the correspondence becomes poor (>40% difference). No doubt this is due to the inadequacy of the fitting procedure in these circumstances; a larger number of Voigt units is required, or, preferably the summation $\Sigma\eta_n$ ought to be replaced by the integral of an appropriate continuous function. More elaborate fitting procedures in which continuous functions are used are described in reference [9].

These results indicate the that the method of incomplete creep can be used to good effect to determine the zero shear viscosity of both unmodified and polymer modified bituminous binders. They also demonstrate that the SHRP parameter can be calculated from a creep test, at least to a first approximation, using a simple numerical procedure.

5. Conclusions

It has been shown that the method of incomplete creep can be used to facilitate the measurement of the creep compliance for both modified and unmodified polymers over a range of temperatures. It has also been shown that a simple numerical procedure can be used to calculate the loss compliance, J'', and hence the SHRP parameter $G^* / \sin \delta$ to a reasonable approximation from creep curves. However the fitting procedure fails for highly elastic materials where the approximation of the continuous retardation spectrum to a finite number of Voigt elements does not hold.

Acknowledgements

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