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Dynamic mechanical response for bituminous mixtures in wide frequency range

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Dynamic mechanical response for bituminous mixtures in wide frequency range

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Abstract. The rheological properties of bituminous mixtures at different test temperatures have been determined by means of a Dynamic Mechanical Analyzer (DMA) via frequency sweep with a relatively wide range between 0.02 Hz and 200 Hz. The measurements were carried out by controlled-strain mode in three-point bending beam test. Continuous spectra were obtained, covering four decades of the frequency scale for phase angle as well as storage and loss moduli with loading frequency. The storage and loss moduli are found to increase as loading frequency increases and temperature decreases. The variation of phase angle with frequency is complicated. Under low test temperature, the phase angle decreases with increasing frequency; under higher temperature, it shows a contrary trend. It is interesting that a peak phase angle appears for these bituminous mixtures in the intermediate temperatures. In addition, the DMA measurement can be used as an effective method to evaluate the viscoelastic properties of bituminous mixtures with fine aggregates.

1. Introduction

Bituminous mixtures are composite of asphalt binder, aggregates, mineral filler, and additives or modifiers if necessary. They are widely used for road construction and bridge pavement. The mechanical properties of bituminous mixtures, whose viscoelastic nature inherits from the asphalt binder, are temperature-, frequency- and time-dependent. The rheological properties of asphalt binders are commonly measured by means of dynamic mechanical methods with a dynamic shear rheometer (DSR) [1, 2] by which the viscoelastic properties are evaluated in terms of complex shear modulus and phase angle. For the viscoelastic property measurements of bituminous mixtures, some standard methods have been developed and extensively used to examine its dynamic mechanical properties by the values of dynamic modulus and phase angle [3]. A sinusoidal (haversine) axial compressive stress is applied to a specimen of asphalt mixtures at a given temperature and loading frequency. The applied stress and the resulting recoverable axial strain response of the specimen is measured and used to calculate the dynamic modulus and phase angle. Generally, the dynamic modulus tests are accomplished at the loading frequencies ranging between 0.1 Hz and 25 Hz. Different traffic speeds can be simulated by different loading frequency. The sinusoidal load change in the test does not fully correspond to the actual nature of the traffic load conditions, no matter how allow for the determination of the rheological properties of the material [4].

A number of research works have been devoted to investigating the complex modulus magnitude and phase angle of bituminous mixtures at different measurement temperatures and frequencies by dynamic modulus tests [4-6]. Typical results of these measurements have been observed that the dynamic modulus increases as loading frequency increases under a constant temperature, while the phase angle shows the opposite trend [5]. Viscoelastic behavior at higher frequency measurements using apparatus of this type is unavailable due to the frequency limitations [3].

Dynamic Mechanical Analyzer (DMA), which is commercially available, such as DMA Q800, produced by TA instruments, is capable of assessing the viscoelastic properties as a function of frequency or temperature, especially for polymers and composites. Such devices can be operated in multiple testing modes, including cantilever, three-point bend, tension, shear and compression deformation modes. It utilizes non-contact, linear drive technology to provide precise control of stress, and air bearings for low friction support. Strain is measured using optical encoder technology that provides unmatched sensitivity and resolution of 1 nanometer. The device allows for frequencies ranging between 0.01 Hz and 200 Hz, covering 4 decades in a relatively broader frequency domain, where as many as frequency points per decade can be taken. Not consequences of a few discrete frequencies, but a continuous spectrum for the complex modulus dependency on the frequency exist through frequency sweep mode. This experiment may be more effective in capturing the viscoelastic behavior. In the case of the Multi-Frequency-Strain mode which can assess viscoelastic response as a function of frequency, the oscillation deformation (strain) amplitude input is held constant at each frequency, and the force (stress) required for this up and down harmonically movement is measured. The ratio of stress to strain, modulus, a function of frequency will be used to characterize the viscoelastic properties of the material.

This paper aims to investigate the dynamic mechanical response of bituminous mixtures over a wide loading frequency range. A Dynamic Mechanical Analyzer (DMA) was adopted, and the three-point bending beam test was performed in Multi-Frequency-Strain mode in which a very small harmonic displacement was applied at each frequency. The test was conducted at various temperatures for the investigation of the effect of temperature on rheological properties.

2. Theoretical background

2.1. Dynamic mechanical analysis (DMA)

For the sake of investigation of the viscoelastic characteristics of material, one may put a small sinusoidal signal excitation on it, and measure its response. In the oscillatory measurements, if the controlled-strain mode is selected, the perturbation is strain, and the corresponding linear response will be the stress with the same angular frequency with that of the input, but leads the applied strain to a phase shift. They can be represented in complex notation as

$$\varepsilon^* = \varepsilon_0 \exp(\mathrm{i}\omega t) \quad (1)$$

$$\sigma^* = \sigma_0 \exp[\mathrm{i}(\omega t + \delta)] \quad (2)$$

where ε_0 and σ_0 are the stain amplitude and stress amplitude, respectively, δ the phase shift, the angular lag between the strain and the stress, t the time in second (s), ω the angular frequency in radians per second (rad./sec), $\omega=2\pi f$, f is the frequency in cycles per second (Hz), and i is the imaginary unit ($\mathrm{i}^2=1$).

Introduction of the phase angle will then give the real and imaginary components of the complex modulus, the ratio of the stress to the strain, which is a complex quantity.

$$E^* = E' + \mathrm{i}E'' = \sqrt{E'^2 + E''^2}\exp(\mathrm{i}\delta) = \left|E^*\right|\exp(\mathrm{i}\delta) \quad (3)$$

$$E' = (\sigma_0/\varepsilon_0)\cos\delta \quad (4)$$

$$E'' = (\sigma_0/\varepsilon_0)\sin\delta \quad (5)$$

$$\delta = \tan^{-1}(E''/E') \quad (6)$$

In the complex plane, the storage modulus E' and loss modulus E'' are respectively the real and imaginary parts of the complex modulus E^*, and $\left|E^*\right|$ is its magnitude. The term tan δ is often called the loss tangent, a measure of the ratio of energy lost to energy stored in an oscillatory test. For an ideal elastic material, a Hookean solid, the stress and strain signals will be in phase at all frequencies, δ=0, whereas for a purely viscous material, a Newtonian fluid, the stress will lead the strain by $\pi/2$ rad at all frequencies. The value of a phase angle for viscoelastic materials is in between 0 to 90 degrees.

A complete presentation of the viscoelasticity theory can be found from Ferry [7], Tschoegl [8] Shaw [9] et al.

2.2. Frequency dependence of viscoelastic response

A possible plot of dynamic response versus logarithmic frequency for a typical viscoelastic solid [10] undergoing simple harmonic excitation is schematically illustrated in Figure 1. The storage modulus exhibits two plateau values, while the loss modulus and phase angle all approach zero at extremely low or high frequencies. In the intermediate frequency range, the storage modulus increases significantly with increasing frequency, however, the loss modulus exhibits a maximum value, as does the phase angle. It also should be noted that the maximum in the loss modulus occurs to the right of that in the phase angle on the frequency scale. For a real solid material, it is by no means necessary that there be a single relative maximum on the curve loss modulus against frequency [10]. Indeed, there may be several such local maxima, each of which is related in some way to rather complicated behavior corresponding to different time constants on a molecular scale [10]. Similarly, there will be several plateaus on the curve of storage modulus versus frequency [7, 11].

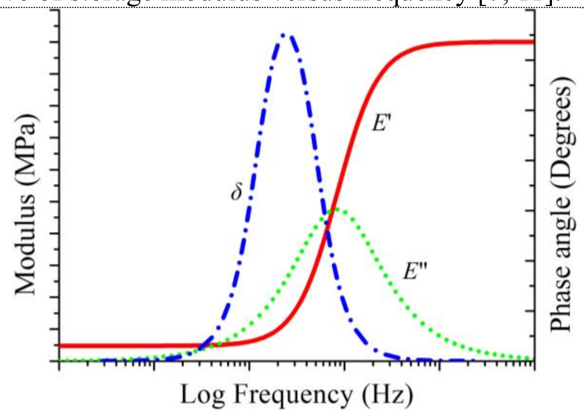

Figure 1. Schematic representation of response versus logarithmic frequency for a typical viscoelastic solid over a broad frequency range.

The storage modulus is a measure of the energy stored and recovered, while the loss modulus is associated with the energy dissipated or lost as heat in sinusoidal deformation. The phase angle, which characterizes the hysteresis, is dependent on the ratio of loss modulus to storage modulus as stated previously. The molecular interpretation of the viscoelastic response exhibited in Figure 1 can be presented as follows [9-12].

At extremely low frequencies, corresponding to long-time scales, reorientation and translation of molecular chain segments occurs more extensively, and the molecular motions can be completely coordinated with the external forces. Hence the viscoelastic solid exhibits rubberlike state, resulting in a modulus decrease and less energy dissipation. As a result, the storage modulus presents a small rubbery plateau with nearly constant value, while the loss modulus approaches zero. In the very low frequency ranges, the strain is approximately in-phase with the stress, that is to say, the storage modulus becomes independent of frequency [12].

However, in the very high frequency ranges, or very short times, there is no sufficient time for substantial molecular reorientation, and the molecular motions essentially cannot coordinate with the external forces, so that the stress cannot be relaxed. Varying intermolecular distances or groups rotating about the bond, being of a relatively high energy, result in a high modulus and low energy dissipated. It corresponds in a sense to the glasslike response. Therefore, it may be expected to have an asymptotic value of glassy modulus that becomes almost independent of frequency and an asymptotic value of zero for the loss modulus. In other words, viscoelastic solids, undergoing very fast or very low processes, respond in an elastic manner at sufficient high frequencies [10].

In the intermediate frequency scales, the chains can move, but the orientation cannot always keep pace with the alternating forces, so energy dissipation takes place, and the material behaves in viscoelastic manner. The loss modulus and phase angle maxima appear at different frequencies.

2.3. *Three-point beam bending test*

For stiff bituminous mixtures specimens, three-point beam bending test is a suitable mode in DMA measurement. The three-point bending beam test is schematically illustrated in Figure 2.

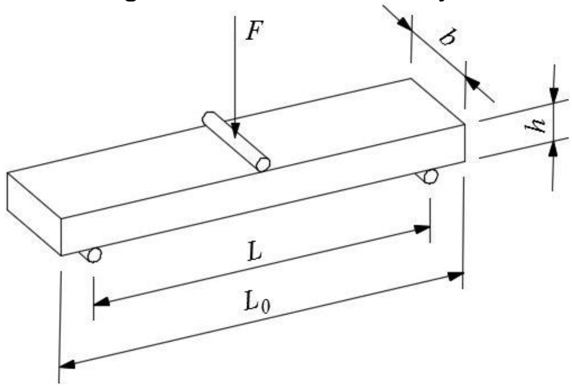

Figure 2. Test geometry for three-point bending beam.

The force is applied at middle of span, then the maximum flexural tensile stress being at the concave and convex surfaces of the beam and the maximum vertical deflection occurred at the mid-span and can be calculated by elementary beam theory.

$$\sigma_{\mathrm{m}} = \frac{3FL}{2bh^2} \quad (7)$$

$$d = \frac{FL^3}{48EI} = \frac{FL^3}{4Ebh^3} \quad (8)$$

$$I = \frac{bh^3}{12} \quad (9)$$

where σ_{m}, the maximum flexural tensile stress, F, the applied load, d, the vertical deflection of beam at mid-span, b, the beam width, h, beam thickness of the cross section, and L, the beam span length, E, the modulus of elasticity, I, the moment of inertia of the cross section, respectively. The modulus of elasticity, E, in equation (8) can be back-calculated using the following equation

$$E = \frac{FL^3}{4bh^3d} \quad (10)$$

The relations between strain ε and stress σ is defined as

$$\varepsilon = \sigma/E \quad (11)$$

By substituting equations (7) and (10) into equation (11), we have the maximum flexural tensile strain ε_{m}

$$\varepsilon_{\mathrm{m}} = \frac{6hd}{L^2} \quad (12)$$

The above formulas are available in textbooks on mechanics of materials. It can be seen that the resulting flexural tensile strain is determined by the vertical deflection d in the same conditions of specimen geometries. For a dynamic experiment, the vertical deflection should be replaced by the amplitude of the oscillatory displacement, while the force should be replaced by the amplitude of the oscillatory force. In the DMA measurement, equation (12) will be used to select appropriate displacement amplitude which must be kept small enough to remain within the linear range of viscoelastic behavior. However, if the selected amplitude is too small, the instrument will not collect data properly.

3. Experimental

3.1. *Materials*

The materials employed for this work are widely used for asphalt pavement in China. Neat asphalt 60/80 penetration grade was used for preparing bituminous mixtures, with the main physical properties listed in Table 1.

Table 1. Properties of used bitumen.

Test	Standard	Unit	Result
Penetration at 25°C	ASTM D5	0.1 mm	68
Softening point	ASTM D36	°C	48.0
Ductility at 15°C	ASTM D113	cm	>100
Viscosity by vacuum capillary viscometer at 60°C	ASTM D2171	Pa·s	232

By considering the maximum size of specimen requirement for bending mode in the DMA test, conventional fine limestone aggregates passing 2.36mm sieve were used to prepare the asphalt mixtures specimens. The gradation can be seen in Table 2.

Table 2. Gradation of used aggregate.

Sieve size (mm)	2.36	1.18	0.6	0.3	0.15	0.075
Cumulative percent passing (%)	100	85.8	53.3	37.6	18.5	12.8

3.2. *Specimen preparation*

The asphalt content was determined as 7.5% by weight of mixture. The asphalt mixtures were rolled in a mold of 300 mm×300 mm×50 mm with a wheel compactor. Thereafter, the sample was cut into some small cuboid blocks, and then cut into final prescribed dimensions, 60 mm in length, 15 mm in width and 5.5 mm in height approximately.

3.3. *Dynamic mechanical analysis (DMA) test*

A TA Q 800 DMA was employed in this work, and the Multi-Frequency-Strain mode of three-point bending beam test was chosen. The mechanical behavior of asphalt mixtures subjected to very small strains can agree with the linear viscoelastic (LVE) theory. The displacement amplitude in the test is required to be kept low enough to ensure remaining in the linear viscoelastic range as well as to ensure that the specimen does not get damaged during the whole frequency sweep process. In this test in which the nominal thickness of specimen h=5.5 mm, beam span length L=50 mm that is determined by the three-point bending clamp, a displacement amplitude d=2 μm was selected, thus the maximum flexural tensile strain ε_m can be calculated by equation (12). It will become a value of 0.00264%, 26.4 micro-strains, which is very small.

The DMA test for asphalt mixtures specimens were carried out on a constant temperature under frequency sweep mode with strain control at displacement amplitude of 2 μm. The specimens were subjected to sinusoidal bending loading, by varying the frequency from the high frequency (200 Hz) to the low frequency (0.02 Hz), where 12 measurements per decade were taken. Eight different temperatures (5, 15, 20, 25, 35, 45, 55 and 65°C) were selected. A conditioning time of more than 30 min was maintained before the commencement of the test, in order to ensure a uniform temperature in the specimen. All the values in the graphs (in Section 4) were averaged by the results of four replicates.

4. Results and discussions

4.1. Modulus
The storage and loss moduli versus loading frequency at different temperatures for bituminous mixtures are plotted with double logarithmic scales, as shown in Figure 3 and Figure 4, respectively.

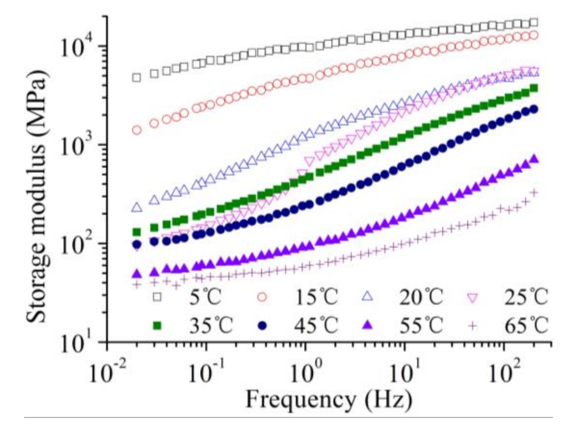

Figure 3. Variation of storage modulus with frequency at different at different temperatures.

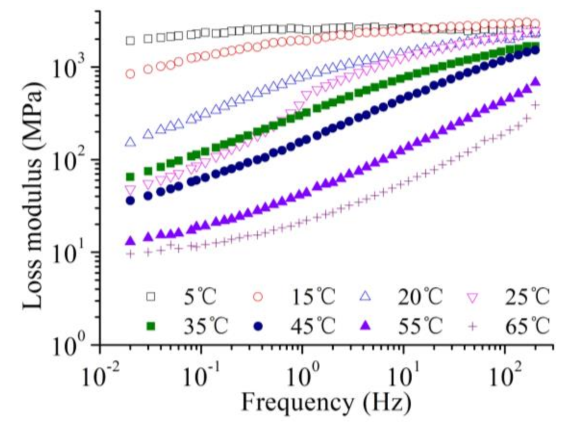

Figure 4. Variation of loss modulus with frequency at different at different temperatures.

The storage modulus is the elastic component relating to the specimen's stiffness, while the loss modulus is the viscous component correlating with the specimen's ability to dissipate mechanical energy. The modulus is a function of frequency as well as temperature. It can be observed that with increasing frequency, the moduli increase at each test temperature. The moduli are reduced as the temperature increases at each experimental frequency. Although the temperatures are different, the patterns for modulus curves are similar but the sensitivity of frequency dependence differs. Higher temperature leads to greater frequency dependence; the loss modulus exhibits a weak frequency dependence taking on an approximate plateau for the specimens of the low temperature of 5°C.

In addition, although the test temperature intervals are small, apparent discrepancy of experimental data can be observed, it may be due to the temperature susceptibility of bituminous mixtures. It also could be attributed to the high sensitivity and accuracy of this type of DMA apparatus.

4.2. Phase angle
The influence of frequency and temperature on the phase angle of bituminous mixtures is shown in Figure 5.

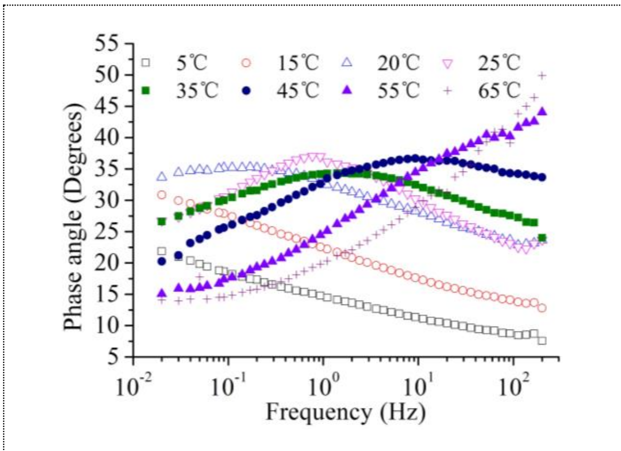

Figure 5. Variation of phase angle with frequency at differeent at different temperatures.

The phase angle, or its tangent value, is a common parameter that provides information on the relationship between the inelastic and elastic components. At the lower temperatures of 5°C and 15°C, the phase angle decreases with increase in frequency. On the contrary, at the relatively high temperatures of 55°C and 65°C, the phase angle shows the opposite trend. However, at the intermediate temperatures of 20°C to 45°C, the phase angle increases first and then decreases with the increase of the loading frequency. Under each test temperature, a maximum value of phase angle arises at a certain frequency. Furthermore, one can see that the frequency at which the phase angle peak occurs shows a right shift with increasing test temperature in the frequency scale.

In this paper, for the bituminous mixtures at relatively low or high temperatures, a maximum in the phase angle is not detectable at all, which is probably due to the insufficient wide frequency range of 0.02 Hz to 200 Hz restricted by the device of this sort. Under low test temperature, the frequency for the occurrence of peak phase angle may lie to the left of low frequency of 0.02 Hz; under high temperature, it may locate at the right of 200 Hz.

As is evident from the discussion above, whether or not the phase angle maximum appears in the oscillatory measurements with frequency sweep depends on the viscoelastic properties of the bituminous mixtures tested and the frequency range. These experimental curves are regarded as only a part of the plot over the entire frequency range described in Figure1.

5. Conclusions

To capture the mechanical response of bituminous mixtures in frequency domain, a Dynamic Mechanical Analyzer (DMA) that widely used in polymer and composite materials has been applied. All the measurements were carried out in frequency sweep mode with wide range of 0.02 Hz to 200 Hz, and three-point bending beam test with strain control was chosen. A variety of temperatures 5, 15, 20, 25, 35, 45, 55 and 65°C were selected to investigate the effect of temperature on viscoelastic properties of bituminous mixtures. In this experimental frequency range, it is shown that the storage and loss moduli increase with increasing frequency for all the test temperatures. However, the observation for variation of phase angle with frequency is complicated. In the case of low or high temperatures, the phase angle decreases or increases with increasing frequency. In the intermediate temperatures, a maximum phase angle appears at a certain frequency whose location is dependent on the test temperature.

Acknowledgements

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