Evaluation of Asphalt-Aggregate Mixture Aging by Dynamic Mechanical Analysis

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Dynamical mechanical analysis (DMA) and other methods of rheological testing have been used to characterize the time- and temperature-dependent responses of viscoelastic materials. A DMA test procedure can determine the complex modulus (E^*), storage modulus (E'), loss modulus (E''), and loss tangent (tan delta). These calculated values can be used to thoroughly characterize an asphalt-aggregate mixture. From tests done over a range of frequencies and temperatures, a master stiffness curve can be made by using the time-temperature superposition principle to transform the data to a standard temperature. From these master curves, complex moduli can be determined at the transformed temperature for frequencies other than those used in the test. At Oregon State University, a semi-closed-loop servohydraulic DMA testing system with controlling software has been developed. An evaluation of three asphalts and two aggregates was undertaken with this new testing system. A range of temperatures from 0° to 40°C and a range of frequencies from 0.01 to 15 Hz was used in the testing program. From the data collected in this program it was possible to differentiate between the different mixtures in their response and to differentiate between long-term aging procedures for some of the mixtures. It was also noted that the DMA rankings of aging susceptibility based on the complex modulus at 1 Hz are similar to the diametral resilient modulus rankings for the same asphalt-aggregate combinations.

The development of laboratory aging procedures to simulate short- and long-term aging of asphalt-aggregate mixtures was undertaken as part of Strategic Highway Research Program (SHRP) Project A-003A at Oregon State University and has been described by Bell et al. (*1–3*). Short-term oven aging (STOA), long-term oven aging (LTOA), and low-pressure oxidation (LPO) were used to investigate the effects of aging on asphalt-aggregate mixtures. Tests such as dynamic mechanical analysis (DMA), diametral resilient modulus, triaxial resilient modulus, and indirect tensile strength were performed on asphalt-aggregate mixtures to quantify the effects of aging.

Diametral and triaxial resilient modulus testing determines only a mixture's elastic response. DMA testing, on the other hand, determines not only a mixture's elastic response, but also its viscous response and phase angle, which Goodrich (*4*) suggests may be stronger indicators of asphalt-aggregate mixture performance.

This paper contains a description of the DMA method used to test specimens that have been conditioned using the STOA and long-term aging procedures outlined by Bell et al. (*1–3*). The results from unaged, short-term aged, and long-term aged specimens are presented.

DYNAMIC MECHANICAL ANALYSIS

Test Method

DMA and other methods of rheological testing have been used to characterize the mechanical behavior of asphalt binders and asphalt-aggregate mixtures (*5–8*). The concept of DMA has been described by Coffman et al. (*9*) and by Sisko and Brunstrum (*10*). DMA can characterize the linear viscoelastic behavior of asphalt binders and mixtures by using the time-temperature superposition method. This behavior is described by a material's time-dependent response (transformed or master curve) and by its temperature-dependent response (shift factors curve) (*5*). The responses measured by DMA in a triaxial mode of testing are complex modulus (E^*), storage modulus (E'), loss modulus (E''), and loss tangent (tan delta), as shown in Figure 1. Papazian (*11*) indicated that these dynamic moduli can provide insights into the time dependence of a material's response and can explain a material's behavior under varying loading rates and durations. For this reason, DMA was used to investigate the change in viscoelastic behavior of asphalt-aggregate mixtures that have undergone accelerated laboratory aging.

Test Procedures

DMA was performed using a modified triaxial mode of testing. A repeated axial load, with no confining pressure, was applied to a specimen using a method similar to the standard test method for the dynamic modulus of asphalt mixtures (ASTM D3497-79). The repeated load was a sinusoidal waveform applied with a sequence of frequencies of 15, 10, 5, 2, 1, 0.5, 0.2, 0.1, 0.05, 0.2, and 0.01 Hz at temperatures of 0°, 25°, and 40°C. The frequency sweep was performed from the highest frequency to the lowest frequency, beginning with the coldest temperature and proceeding to the warmer temperatures. Load and vertical deformation were monitored during

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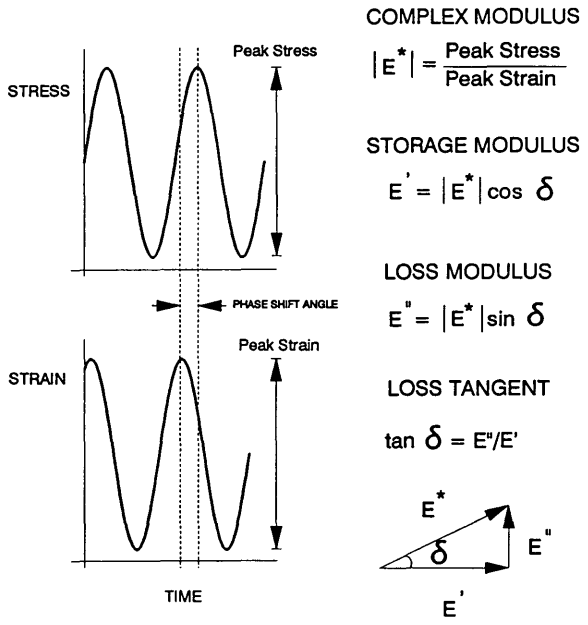


FIGURE 1 Dynamic mechanical analysis [after Goodrich (*8*)].

the test. Load was measured by a load cell at the bottom of the specimen. Vertical deformation was measured by two linear voltage differential transducers (LVDTs) attached to the side of the specimen with a set of yokes.

The yokes were separated by four 2-in. spacers before they were glued with cyanoacrylate adhesive to the specimen. The glue was allowed to set for 10 min at room temperature (25°C) before the specimen was cooled to 0°C in an environmental cabinet. A specimen with an imbedded thermocouple was also placed in the cabinet as a control. When the control specimen reached 0°C, the other specimens in the cabinet were ready for testing. A frequency sweep on a particular specimen takes about 25 min at each temperature. A set of six specimens can be tested at all three temperatures in one testing day (12 hr).

After the test at 0°C was completed, the specimen was placed in another environmental cabinet set at 25°C. A control specimen was again used to monitor the temperature of the other specimens in the cabinet. As before, once the control specimen reached the next test temperature, the other specimens were ready for testing. During the test program, the load cell and LVDTs were calibrated at various temperatures. It was found that the calibration factors were constant within the range of testing temperatures. This test was nondestructive, with the total recoverable deformation limited to 200 in. at both the lowest frequency (0.01 Hz) and the highest test temperature (40°C). The test was performed by adjusting the load to produce a recoverable strain of 25 strain at 1 Hz. The stress required to induce the 25 strain at 1 Hz was used as the applied stress throughout the frequency sweep test to ensure that the strain level did not exceed 100 strain at any other frequency or temperature. A procedure to control the strain at, for example, 100 strain would be preferable but more difficult to achieve. The collected data were processed to generate dynamic moduli and phase angles.

The test was performed on an MTS servohydraulic semi-closed-loop control system. The data acquisition and analysis were performed on a high-speed 486 personal computer. The computer software controlled the MTS machine during the frequency sweep and saved the data to a file. The data were processed to generate the dynamic moduli and phase angles.

Test Analysis

The fundamental material responses obtained from DMA, characterizing the viscoelastic behavior of the materials as a function of frequency (loading time) and temperature, are the dynamic moduli: the complex modulus, the storage modulus, and the loss modulus. The loss tangent is calculated from the ratio of the loss modulus to the storage modulus. The dynamic modulus results are transformed to a standard temperature, in this case 25°C, by using the time-temperature superposition principle to create a master curve. The general process of transforming data to develop the master curve is shown in Figure 2 [after Stephanos (*12*)].

Figure 3 shows a master curve constructed from DMA performed on a short-term-aged specimen. Data collected at 0°C have been shifted to the right into higher-frequency ranges, whereas data at 40°C have been shifted to the left into lower-frequency ranges. The points thus line up to make a smooth S-curve that includes frequencies outside the original test range. A unique phase shift curve was developed for each specimen describing the amount of shift (α) for each test temperature. The curve is used to produce transformed plots for each DMA parameter (as shown in Figure 2), for example, complex modulus or phase angle, for any temperature within the range tested. In this study, transformations to 25°C were used.

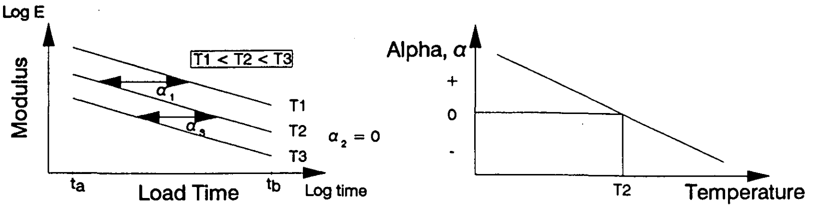
EXPERIMENT DESIGN

Aging Methods

Five different aging treatments were considered for this experiment. This experimental program is part of a larger test program to compare the results of aging of asphalt (SHRP Project A-002A) with the results of aging of asphalt-aggregate mixtures (SHRP Project A-003A). The full test program has been described by Bell and Sosnovske (*13*). One short-term and four long-term aging treatments were considered, as follows:

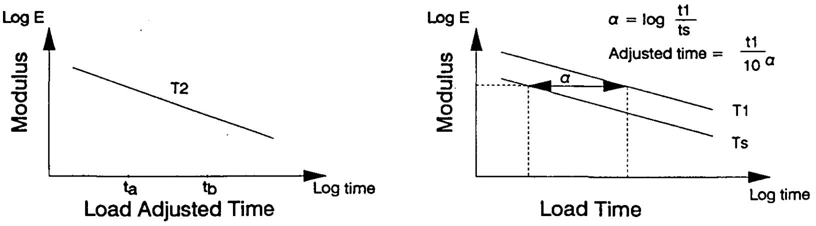
1. No aging,
2. STOA for 4 hr at 135°C,
3. LTOA for 5 days at 85°C,
4. LTOA for 2 days at 100°C,
5. LPO for 5 days at 60°C, and
6. LPO for 5 days at 85°C.

STOA was performed on loose asphalt-aggregate mixtures at 135°C for 4 hr. After being aged, the mixtures were compacted at an equiviscous temperature of the unaged asphalt corresponding to a viscosity of 6 poises (665 ± 80 cSt). The mixing and compaction procedures followed protocols established by the SHRP A-003A team, which in turn are based



a. Modulus vs Time in Log-Log space.

b. Alpha vs Temperature.



c. Master Curve in Log-Log space.

d. Calculation of Adjusted Time.

FIGURE 2 Procedure for transforming modulus data to the master stiffness curve [after Stephanos (*12*)].

on the methods used to prepare Hveem specimens (ASTM D1560-81a and D1561-81a). All of the mixture specimens were cylinders 4 in. high by 4 in. in diameter.

Long-term aging was performed at different temperatures and for different aging periods in order to investigate the effects of temperature and duration on the severity of the aging of the asphalt-aggregate mixtures. Specimens were subjected to the STOA procedure before undergoing either the LTOA or LPO aging treatment. LTOA was performed for 5 days at 85°C or for 2 days at 100°C on different specimens. The specimens were conditioned in a forced-draft oven.

LPO was also performed at two different temperatures, 60° and 85°C, but for only one duration, 5 days. The specimens were sealed in a modified triaxial cell that was subsequently submerged in a water bath to control temperature. Oxygen was passed through the specimen at a flow rate of 4 ft^3/hr.

The unaged specimens were prepared to compare with asphalt-aggregate mixtures that had been subjected to various aging methods. The unaged specimens were compacted immediately after being mixed.

Evaluation Methods

The tests used in addition to DMA to evaluate the effects of aging were the resilient modulus (ASTM D4123) and indirect tensile (ASTM D4123) tests.

An extensive program of testing was conducted using the resilient modulus approach. This program is reported in detail in another paper in this Record (Sosnovske et al.). The resilient modulus was determined using an indirect tensile mode and a triaxial mode of testing. The repeated load applied to the specimen was a haversine waveform. The load was applied in the vertical diametral plane of a cylindrical specimen of asphalt mixture. The recoverable or resilient horizontal deformation of the specimen was measured and with an assumed

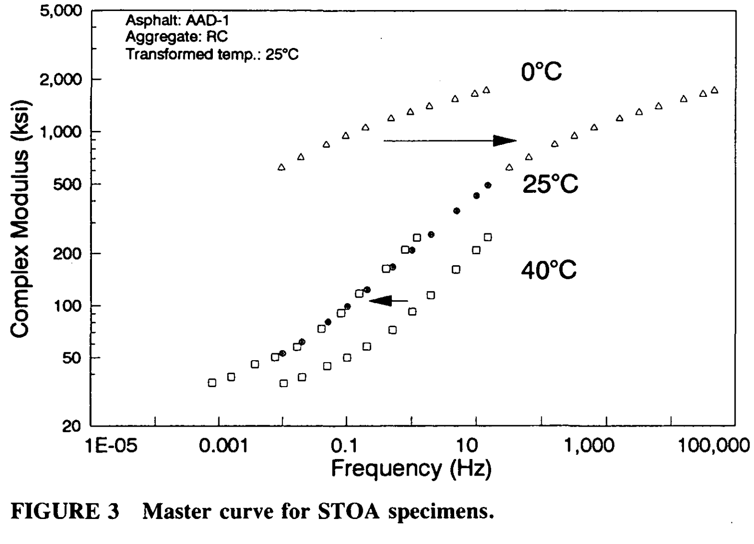


FIGURE 3 Master curve for STOA specimens.

Poisson's ratio of 0.35 was used to calculate a diametral resilient modulus after many load repetitions. Triaxial resilient modulus testing was performed by applying repeated compressive stress with no confining pressure on a specimen using a haversine waveform. The stress was applied uniaxially on a cylindrical specimen of asphalt mixture and the recoverable or resilient axial deformation was measured after many repetitions. The triaxial resilient modulus was the ratio of the repeated axial stress to the recovered axial strain.

Test Program

Two specimens from each combination of asphalt and aggregate were used for each long-term aging treatment. STOA at 135°C for 4 hr on loose mixtures was performed on all long-term-aged specimens. The asphalts and aggregates used for this test program were selected from a more extensive set in the SHRP Materials Reference Library that had been used in companion studies (*1–3*, *13*).

RESULTS

The data presented here were obtained from tests on aggregates RC and RH mixed with asphalts AAD-1, AAF-1, and AAM-1. The test specimens were compacted using a nominal air void target of 8 percent. The air void percentage for each specimen is shown in Table 1.

Figure 4 shows typical complex modulus and phase angle data that have been shifted on the transformed frequency scale. The complex modulus data were fitted using a modified hyperbolic secant function to produce an equation with parameters that describe the master curve's behavior. The phase angle data were fitted using a fourth-power parabolic equation. Figure 5 shows a typical plot of fitted data for a mixture different from that shown in Figure 4. The plots for unaged specimens were obtained from DMA performed on three specimens. The plots for STOA specimens were obtained from tests on a pair of specimens that were subsequently long-term aged and retested. Since four long-term aging methods were evaluated, a total of eight specimens were aged for each asphalt-aggregate combination. In the interest of space, only data for long-term aging at 85°C are shown here.

Table 1 shows the diametral resilient modulus aging ratios for each aging treatment. The aging ratio is defined as the ratio of an aged specimen's diametral resilient modulus to its unaged resilient modulus. Aging ratios are plotted in Figure 6 to show the diametral resilient modulus rankings of asphalt-aggregate combinations for each aging treatment. A complete discussion on the presentation of resilient modulus data can be found in the companion paper in this Record by Sosnovske et al. The complex moduli at frequencies of 0.001, 1, and 1,000 Hz are shown in Table 2. These values are obtained from the master curves transformed at 25°C. Table 3 shows the DMA complex modulus ratios of aged specimens to unaged specimens for each aging treatment at frequencies of 0.001, 1, and 1,000 Hz. Figure 7 shows the complex modulus rankings of asphalt-aggregate combinations.

DISCUSSION OF RESULTS

Typical data obtained by Christensen and Anderson (*5*) from DMA results on asphalt binders are shown in Figure 8. A master curve for a typical asphalt binder shows that the complex modulus approaches a limiting elastic value at high frequency at about 1 GPa (145 ksi). The modulus decreases monotonically as the frequency is reduced. The curve at very low frequencies usually slopes at a 1:1 ratio, which indicates that viscous flow has been reached.

Master curves of complex modulus versus frequency for asphalt mixtures (e.g., Figures 4 and 5) were constructed that showed similar trends to the master curves for asphalt binders at high frequencies. The master curves show that the complex modulus at high frequencies generally approaches a limiting value of about 5,000 ksi after long-term aging treatments. As the frequency is reduced, the complex modulus decreases from the highest frequency to the lowest frequency following the S-curve. It appears as though each complex modulus curve could be modeled by a linear portion in the middle frequencies, showing viscous response, and by curved portions at high and low frequencies, where elastic behavior is approached. The tendency toward elastic behavior at low frequencies is very apparent for the unaged specimen and indicates that the aggregate is dominating the response. However, for the short-term-aged specimens, the complex moduli are higher and the

TABLE 1 Summary of Voids and Resilient Modulus Ratios for Short-Term and Long-Term Aging

Aggregate	Asphalt	Aging Ratio			
		Short-Term Oven	Long-Term Oven @ 100 C	Long-Term Oven @ 85 C	Low Pressure Oxidation @ 85 C
RC	AAD-1	1.59	3.69	3.43	3.63
	AAF-1	1.34	2.01	1.90	2.18
	AAM-1	1.35	2.47	2.41	2.42
RH	AAD-1	1.72	4.03	2.84	2.21
	AAF-1	1.26	1.67	1.41	1.45
	AAM-1	1.36	1.97	1.67	1.91

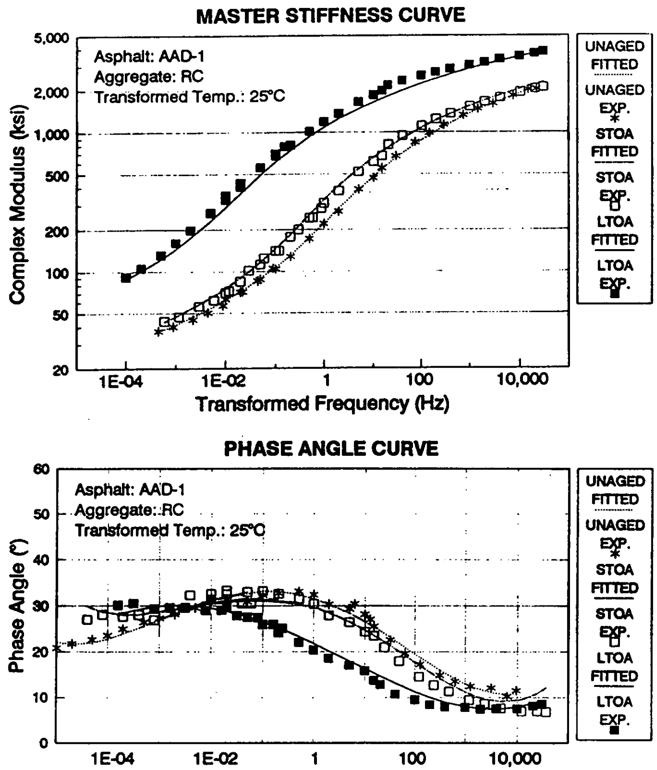


FIGURE 4 Typical experimental and regression data for master and phase angle curves.

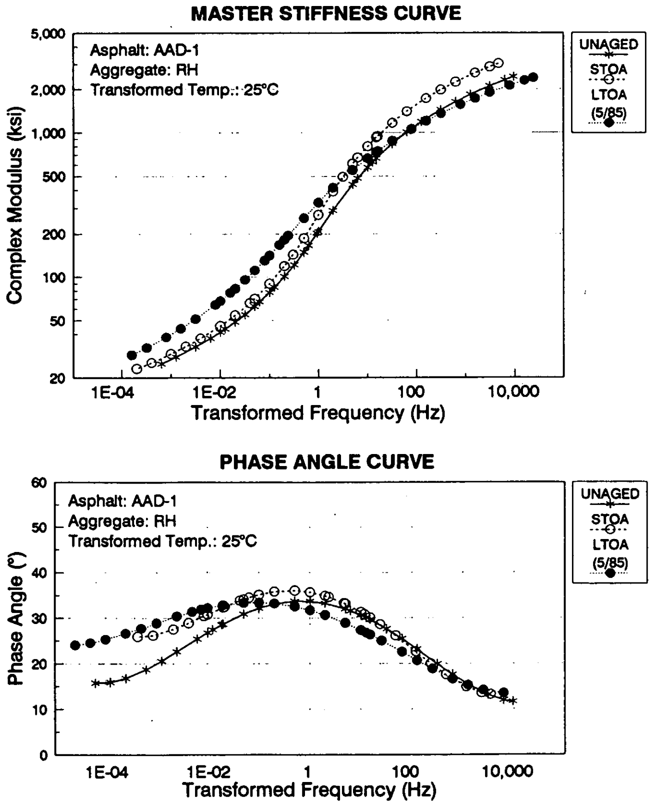


FIGURE 5 Master curve and phase angle curve for asphalt AAD-1 and aggregate RH.

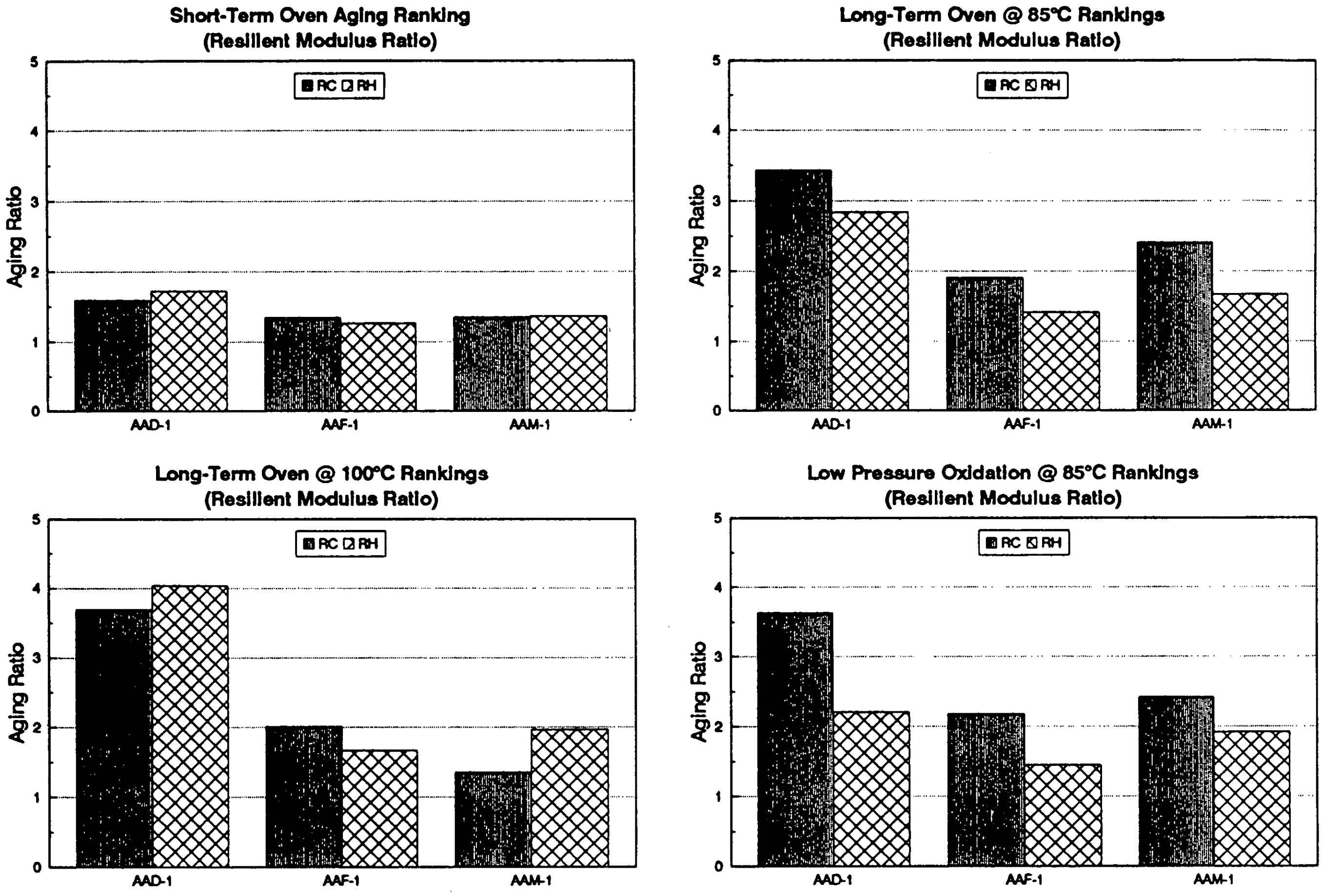
elastic response is less evident at low frequencies. The complex moduli are even higher for long-term aging curves, depending on the type of treatment, and have similar trends to the STOA curves.

The complex modulus data suggest that the unaged specimens at low frequencies, where the test temperature is high, correspond to the lower limit of viscous (flowing) behavior of the asphalt-aggregate mix and that they approach the conditions where the aggregate tends to dominate the material response. After undergoing STOA, the complex modulus of the asphalt-aggregate mixtures increases. At low frequencies and medium test temperature, the master curves slope to a 1:1 ratio, which indicates that the mixtures are undergoing viscous response. Similarly, the master curves for long-term-aged specimens show the same trend with higher complex moduli at lower frequencies. At high frequencies and low test temperature, the master curve starts to approach an elastic asymptote as the asphalt stiffness approaches elastic behavior.

Plots of phase angle versus frequency (e.g., Figures 4 and 5) show that the curves peak in the frequency range of 0.01 to 1 Hz. Similar peaks are found in the data presented for modified asphalt mixtures (*7*). This similarity confirms that at an intermediate temperature or frequency, the asphalt-aggregate mixture is more viscous than at high or low frequencies where either the asphalt or aggregate dominates the elastic response. At high frequency (or low temperature), the phase angle is small, indicating that the asphalt behaves like an elastic material. According to Lazan (*14*), this is known as the "glassy" region where various types of molecule mobilities are "frozen-in." As the frequency decreases (or the temperature increases), the phase angle reaches a maximum. The asphalt behaves like an elastic material at high frequency, gradually changes to a viscoelastic material, and continues to change into a viscous material as the frequency decreases (or the temperature increases). After the phase angle maximum, the phase angle gradually decreases to a minimum where the mixture is again in the viscoelastic phase, even though the asphalt viscosity continues to decrease. The aggregate appears to dominate the mixture property at this point.

The peak phase angles for unaged, short-term-aged, and long-term-aged specimens are less than 45 degrees. After short-term aging, the phase angle peak is lower and shifted to the left, indicating that the specimen is stiffer and that the modulus has increased. Similarly, the phase angle peak for long-term-aged specimens tends to be flatter and lower than the short-term phase angle peak, depending on the aging treatment. The long-term peak is shifted even more to the left, which shows that the specimen has become even stiffer. Hence, the phase angle curves are very good indicators of mixtures' becoming more viscous with aging. It may be feasible to determine limiting values of phase angle maxima or minima in order to control cracking of asphalt pavements.

At high temperature or low frequency, Table 2 shows that the combinations with aggregate RH have a lower complex modulus and, at low temperature or high frequency, have a higher complex modulus than the combinations with aggregate RC. The RH combinations also have higher phase angle values that peaked at higher frequencies. Phase angles that are too high might be associated with pavement rutting. These high phase angles indicate that these mixes are more susceptible to modulus change with either frequency or temperature.


Short-Term Oven Aging Ranking (Resilient Modulus Ratio)

Long-Term Oven @ 85°C Rankings (Resilient Modulus Ratio)

Long-Term Oven @ 100°C Rankings (Resilient Modulus Ratio)

Low Pressure Oxidation @ 85°C Rankings (Resilient Modulus Ratio)

FIGURE 6 Diametral resilient modulus rankings of asphalt-aggregate mixtures for each aging treatment.

TABLE 2 Complex Modulus Data in Kips per Square Inch Selected at Frequencies of 0.001, 1, and 1,000 Hz

Aggregate	Asphalt	Frequency	Complex Modulus (ksi)				
			Unaged	Short-term Oven @ 135 C	Long-Term Oven @ 100 C	Long-Term Oven @ 85 C	Low Pressure Oxidation @ 85 C
RC	AAD-1	0.001 1 1000	42 280 1400	50 330 1620	105 670 2180	155 1020 2900	195 1190 3300
	AAF-1	0.001 1 1000	69 710 2320	100 890 2650	180 1250 3050	210 1450 3300	330 1850 4000
	AAM-1	0.001 1 1000	50 470 1450	85 610 1950	160 960 2350	130 800 2100	215 1200 2700
RH	AAD-1	0.001 1 1000	35 190 1680	42 280 2000	65 520 2420	48 340 2050	55 430 2150
	AAF-1	0.001 1 1000	60 740 2750	70 890 3000	120 1000 3100	140 1100 3200	170 1300 3400
	AAM-1	0.001 1 1000	50 495 1950	68 550 2200	70 650 2350	79 770 2500	125 1080 3050

TABLE 3 Complex Modulus Ratios Selected at Frequencies of 0.001, 1, and 1,000 Hz

Aggregate	Asphalt	Frequency	Complex Modulus Ratio				
			Unaged	Short-Term Oven @ 135 C	Long-Term Oven @ 100 C	Long-Term Oven @ 85 C	Low Pressure Oxidation @ 85 C
RC	AAD-1	0.001	1.0	1.2	2.5	3.7	4.6
		1	1.0	1.2	2.4	3.6	4.3
		1000	1.0	1.2	1.6	2.1	2.4
	AAF-1	0.001	1.0	1.4	2.6	3.0	4.8
		1	1.0	1.3	1.8	2.0	2.6
		1000	1.0	1.1	1.3	1.4	1.7
	AAM-1	0.001	1.0	1.4	1.4	1.6	2.5
		1	1.0	1.1	1.3	1.6	2.2
		1000	1.0	1.1	1.2	1.3	1.6
RH	AAD-1	0.001	1.0	1.2	1.9	1.4	1.6
		1	1.0	1.5	2.7	1.8	2.3
		1000	1.0	1.2	1.4	1.2	1.3
	AAF-1	0.001	1.0	1.2	2.0	2.3	2.8
		1	1.0	1.2	1.4	1.5	1.8
		1000	1.0	1.1	1.1	1.2	1.2
	AAM-1	0.001	1.0	1.7	3.2	2.6	4.3
		1	1.0	1.3	2.0	1.7	2.6
		1000	1.0	1.3	1.6	1.4	1.9

Note: Ratio calculated by dividing with unaged complex modulus.

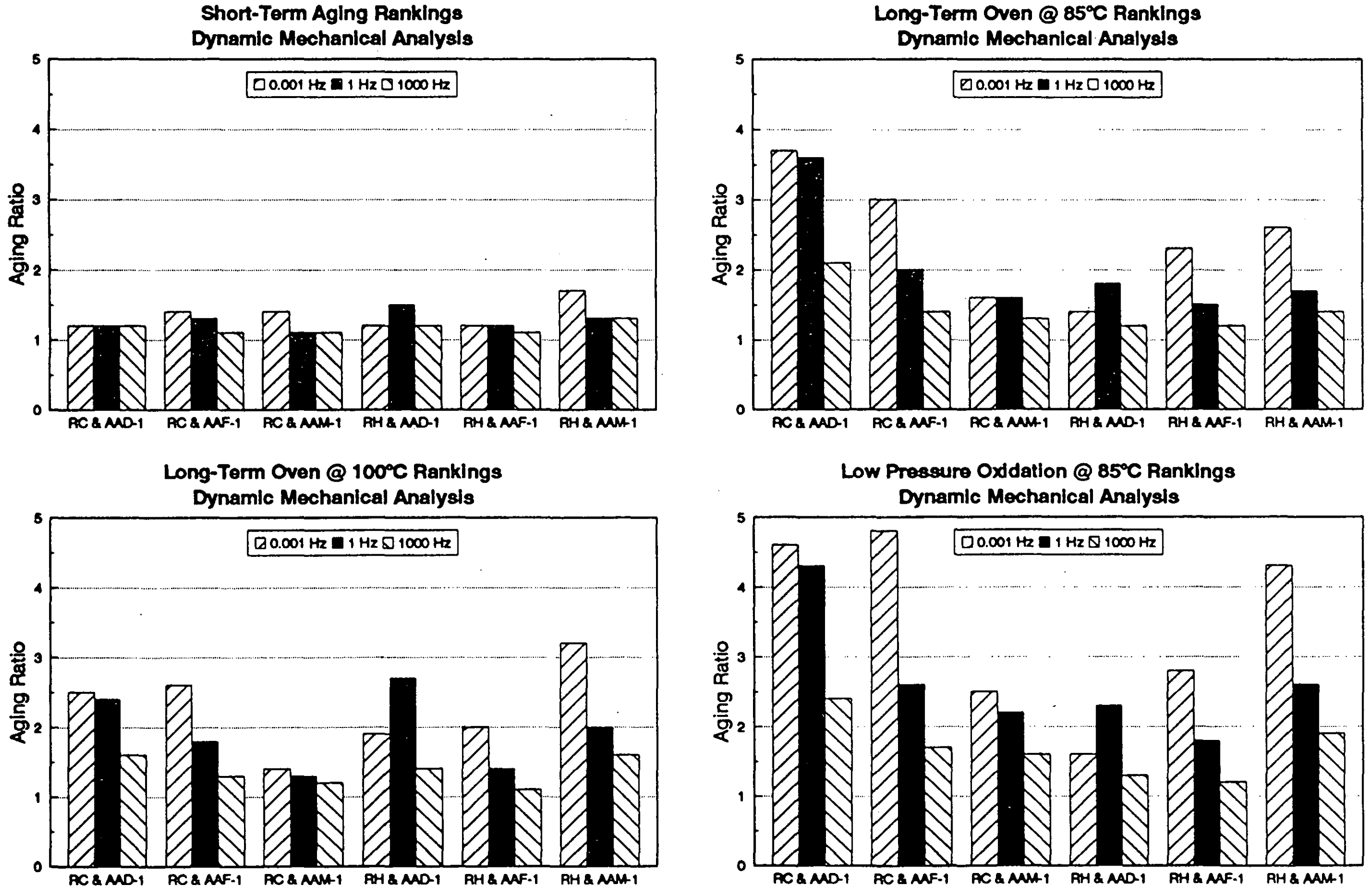


FIGURE 7 Complex modulus rankings of asphalt-aggregate mixtures for each aging treatment.

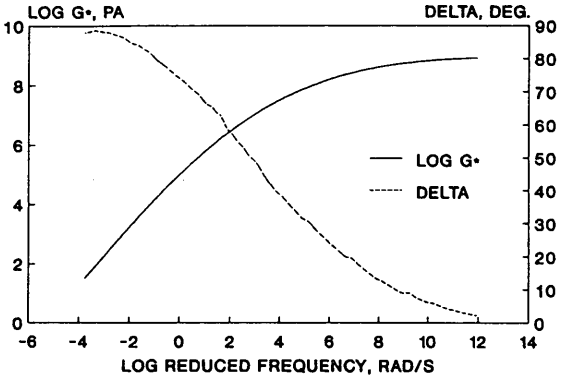


FIGURE 8 **Master curve for asphalt AAB-1 (*5*).**

The data in Tables 2 and 3 also show that mixtures with aggregate RC show a greater change in complex modulus after short-term and long-term aging, especially at high temperature (or low frequency), than do mixtures with aggregate RH. The mixtures become stiffer in the low-frequency region, which suggests that the mixtures have undergone more aging after being long-term oven aged for 5 days at 85°C. Similarly, the phase angle curves show that the phase angle peaks for RC mixtures shifted more to the left than they did for RH mixtures. This shows that the viscous component of RC mixtures changed more than that of RH mixtures after short-term and long-term aging treatments. The mixtures with aggregate RH have higher peak phase angles than mixtures with aggregate RC, which suggests that RH mixtures are more viscous. Figure 6 shows the diametral resilient modulus rankings of asphalt-aggregate mixtures for each aging treatment. Further discussion on evaluating the effects of aging by diametral resilient modulus may be found in the companion paper by Sosnovske et al. in this Record. Figure 7 shows the complex modulus rankings of asphalt-aggregate mixtures for each aging treatment. The complex moduli at 0.001, 1, and 1,000 Hz were obtained from the master curve plots of unaged, short-term, and long-term aging. The ratios were calculated by dividing the aged complex modulus into the unaged complex modulus. These ratios were compared with the ratios calculated using diametral resilient modulus data. It was found that the DMA plots at 1 Hz have the same rankings as those plotted for the diametral resilient modulus for all aging treatments. However, the plots at 0.001 and 1,000 Hz vary for each treatment. This variation suggests that DMA can indicate the behavior of each asphalt-aggregate mixture at different test temperatures in terms of each mixture's susceptibility to aging.

CONCLUSIONS

The following conclusions were reached:

1. A method of determining dynamic mechanical properties of asphalt-aggregate mixtures has been developed. It is feasible to test several specimens in one day (8 hr) and obtain valuable data regarding frequency, temperature, and aging susceptibility of mixtures.
2. The short-term-aged specimens are consistently stiffer than the unaged specimens for all asphalt-aggregate combinations. The long-term specimens are consistently stiffer than the short-term and unaged specimens for the aggregate RC combinations, but this was not the case for some other combinations because of scatter in the data.
3. LPO for 5 days at 85°C is the severest treatment among the evaluated long-term aging treatments.
4. DMA rankings based on the complex modulus at 1 Hz are similar to the diametral resilient modulus rankings for the evaluated asphalt-aggregate combinations.

RECOMMENDATIONS

The following recommendations were made:

1. DMA may be an excellent indicator of asphalt-aggregate mixture susceptibility to rutting or cracking before and after aging. A mathematical model to quantify the master curve of asphalt-aggregate mixtures in terms of a few parameters is needed.
2. Measurements to include the Poisson's ratio in the DMA test would be desirable in order to investigate the changes in lateral and vertical deformation of asphalt-aggregate mixtures after aging.
3. An option to include confining pressure during the DMA test should be considered in order to simulate field conditions.
4. Additional test temperatures to allow for isochronal plots (*5*) are desirable in order to investigate asphalt-aggregate mixture behavior over a wider range of test temperatures.

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