Nonlinear oscillatory shear tests applied to bitumen: analysis and application of large amplitude oscillatory shear (LAOS)

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ABSTRACT

The characterization of bitumen binder fatigue properties has been a difficult challenge since the introduction of the Superpave system in the early 90's. Originally, rheological testing was performed in the linear viscoelastic region, but was found not necessarily adequate to show the impact of modified binders. Recently, researchers at the University of Madison-Wisconsin considered nonlinear region conditions to develop the Linear Amplitude Sweep (LAS) test. They showed the potential of such conditions to differentiate binders with respect to modification. In this study we have tried to go one step further by characterizing the rheological behavior of bitumen in the nonlinear viscoleastic region by an oscillatory deformation protocol known as large amplitude oscillatory shear (LAOS). To date, there has been little application of LAOS to characterize the nonlinear viscoelasticity of unmodified and modified bitumen. The common practice has been to apply the "viscoelastic moduli" corresponding to the first harmonic Fourier coefficients G1' (ω), G1'' (ω). However, in many cases that can be misleading in describing the nonlinear phenomena. The focus of this paper is to demonstrate the application of LAOS to the LAS test and point out some limitations in the way the LAS test currently analyzes oscillatory shear in the nonlinear regime using just the first harmonic. The framework of our LAOS analysis is in terms Lissajous curves and Fourier series and transforms.

Keywords: Fatigue Cracking, Performance testing, Rheology

1. INTRODUCTION

Fatigue cracking is one of the most important damage modes of asphalt pavements and yet one of the most difficult to comprehend and to assess. In the US, the SHRP program made substantial improvement on the binder side by introducing a rheological parameter to capture binder fatigue resistance based on the measure of the binder dissipated energy under shear in the linear domain. It has been reasonably efficient for assessing unmodified bitumen working under low strain, but not so well for modified binders.

More recently, the Linear Amplitude Sweep (LAS) bitumen fatigue test was developed by H. Bahia and coworkers at the University of Wisconsin, USA, to overcome these shortcomings. The LAS test has been proposed as an alternative to the time sweep test used in the standardized Superpave dissipated energy measurement. The major difference between the time sweep and LAS test is that the time sweep is performed with constant strain amplitude, whereas with the LAS test the strain amplitude is systematically increased to accelerate damage. The LAS test was adopted by AASHTO (TP-101-12 *Test for Estimating Fatigue Resistance of Asphalt Binders Using the Linear Amplitude Sweep*) as a provisional method in 2012. Other studies have suggested that the damage in binders tested at high strains over a comparatively limited number of cycles does not necessarily relate to fatigue damage (Gauthier et al, 2010). However, the tests presented in this paper use different metrics to assess potential fatigue resistance as discussed below and therefore warrant additional investigation.

The test temperature is not specified in TP 101-12-UL. There is a recommendation that the test temperature should be determined so as to insure that damage during the test only manifests as fatigue cracking. When testing above a certain temperature, flow failure dominates; similarly, testing below another certain temperature, adhesive failure between the DSR plates and asphalt specimen or thermal cracking dominates. The allowable range of the complex shear modulus, G*, to insure fracture is 10 to 50 MPa. The selected complex modulus within this range is sometimes referred to as the iso-modulus, and the temperature at which it is reached is referred to as the iso-modulus temperature.

There is another hypothesis, however, that the test temperature should be selected based on the Superpave intermediate Performance Grade (PG) temperature for the particular climate involved, similar to the test temperature selection for low and high temperature Superpave bitumen testing.

This paper presents the bitumen fatigue test results on four bitumens used at the Arizona (AZ) validation site constructed in 2001 under the Asphalt Research Consortium (ARC) cooperative agreement between the Western Research Institute and the Federal Highway Administration. In this study, two different fatigue tests were used: the Linear Amplitude Sweep (LAS) Test and a Large Amplitude Oscillatory Shear (LAOS) Test. The bitumens were first oxidatively aged according to the standard RTFOT and PAV protocols. For this study, rather than determine an iso-modulus test temperature for each of the four RTFO/PAV bitumens, they were tested at 28°C, which is the appropriate Superpave intermediate test temperature for the Arizona test site climatic zone. This temperature selection method was used for the sake of simplicity in this preliminary study although using the iso-modulus temperature selection method would certainly compare the binders in closer stiffness conditions.

The most common type of LAOS experiment is strain-controlled where a sinusoidal strain, $\gamma(t)$, is applied, and the oscillatory stress response, $\sigma(t)$, is measured:

$$\gamma(t) = \gamma_o \sin(\omega t)$$

$$\sigma(t) = \sigma_o \sin(\omega t + \delta)$$

where γ_o is strain amplitude and the phase angle between the imposed strain sine wave and stress response sine wave is δ. G” is the viscous (out-of-phase) component and G’ is the elastic (in-phase) component of the complex shear modulus, G*:

$$G^*(\omega) = G'(\omega) + iG''(\omega)$$

Figure 1 illustrates the three types of behaviors that can occur depending on the phase angle in the linear regime. As the phase angle approaches zero, the material response is primarily elastic and forms a straight line. As the phase angle approaches 90 degrees, the response is primarily viscous and forms a circle. When the phase angle lies between 0 and 90 degrees, the curve is elliptical. The elliptical stress-strain curves are called Lissajous curves.

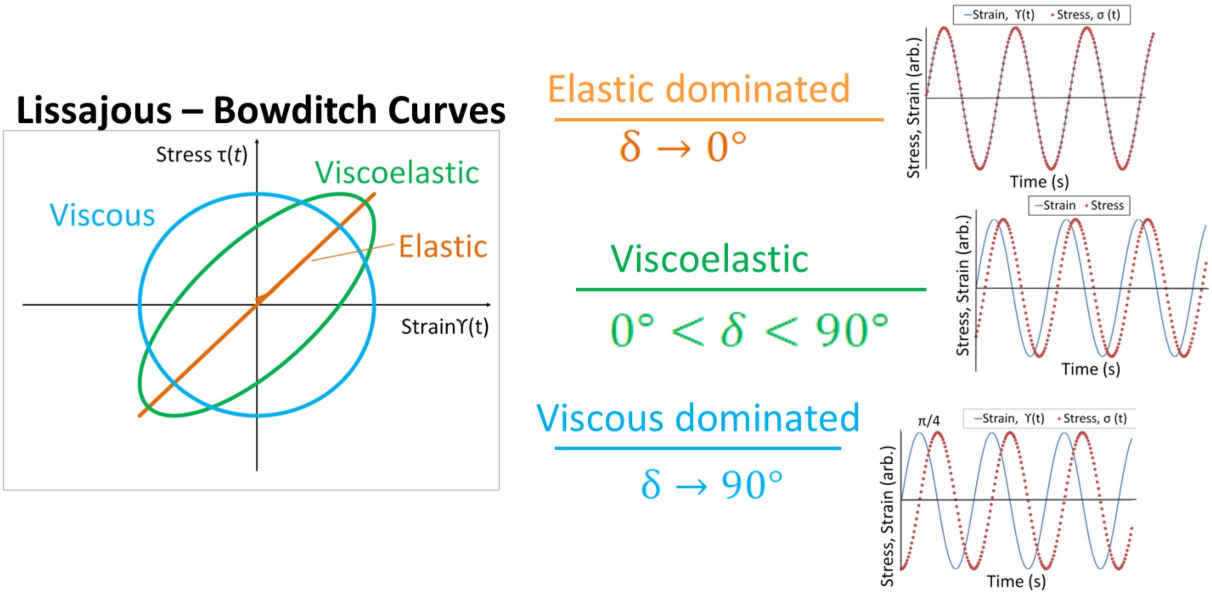

Figure 1. Linear viscoelasticity (small amplitude oscillatory shear (SAOS).

As the strain amplitude is increased beyond the linear viscoelastic regime, it becomes difficult to interpret the dynamic moduli because the stress wave response becomes distorted and no longer completely sinusoidal. Because of this distortion in the nonlinear regime, multiple dynamic moduli corresponding to the odd harmonics in the frequency domain arise. For this reason, typical rheological theory, which takes into account only the linear viscoelastic region and therefore only the first harmonic moduli, is not completely accurate.

The LAOS test protocol was specifically drafted for use in this study; thus it is expected that it will continue to evolve as understanding of the phenomena observed therein does. The LAOS test is similar to the LAS test in that it applies a strain ramp to a sample at a predetermined intermediate temperature and at constant shear rate. This marks the extent of the similarities between the two test protocols, which are described in greater detail in the experimental section.

2. EXPERIMENTAL

The testing was performed using a Malvern Kinexus dynamic shear rheometer (DSR). Eight mm diameter plates and a 2 mm gap were used for all testing. The LAS test was performed in general accordance with TP 101-12-UL.

The four AZ samples used for this study are named AZ1-1, AZ1-2, AZ1-3, and AZ1-4. Since all four samples were subjected to RTFO and PAV lab ageing, their names are abbreviated for simplicity. Table 1 lists the samples used in this study and their abbreviated names, along with their complex moduli at the start of the LAOS test sweep.

Table 1. WRI-3 Arizona bitumen samples tested using the LAS and LAOS tests.

Full Sample Name	Simplified Sample Name	Superpave PG grade	G* @ 1Hz @ 0.01% strain @ 28°C (Pa)
AZ1-1_ RTFO/PAV	AZ1-1 RP	76-16	3.98E+06
AZ1-2_ RTFO/PAV	AZ1-2 RP	76-22	3.09E+06
AZ1-3_RTFO/PAV	AZ1-3 RP	76-16	6.86E+06
AZ1-4_RTFO/PAV	AZ1-4 RP	76-16	8.90E+06

2.1 LAS Test Procedure and Analysis

There are four principle steps in performing the LAS test:
(1) Determine the Superpave intermediate test temperature;
(2) Perform a frequency sweep (0.2 to 30 Hz) at 0.1% strain at the intermediate test temperature;
(3) Perform the strain ramp function at 10Hz at the intermediate test temperature;
(4) Analyze the frequency sweep and strain ramp results to calculate the Viscoelastic continuum damage (VECD) fatigue resistance number.

2.1.1 Determine Superpave Test Temperature

The intermediate test temperature was determined using the standard Superpave methodology for the Arizona climate. The test temperature was determined to be 28°C for all four samples used in this study. This means they have very similar loss moduli of not more than 5 MPa at 10rad/sec at this temperature. This is fair to assume this temperature is close to an iso modulus temperature then. However, at the Superpave intermediate temperature, the samples have moduli between 3 and 9MPa, and thus are slightly outside the lower bound of 10MPa suggested in the iso-modulus method. Thus, there is a risk of deformation during the LAOS test that may not be classified as pure fatigue behavior. An attempt was made to avoid this by running the four samples at the intermediate temperature -10°C (18°C) but unfortunately all four samples suffered total failure at strains inferior to 3%, so reliable data could not be obtained.

2.1.2 Frequency Sweep

The frequency sweep is performed at 0.1 ± 0.01 percent strain over a range of frequencies from 0.2–30 Hz. Data are sampled at 12 frequencies. The frequency sweep is used to determine a damage analysis "alpha" parameter and is performed at the selected intermediate temperature. The alpha parameter is fundamentally related to the ability of the material to relax and thus resist fracture.

The frequency sweeps performed for the four AZ bitumens are shown in Figure 2. They display similar frequency susceptibility except for AZ1-1 RP which appears less susceptible.

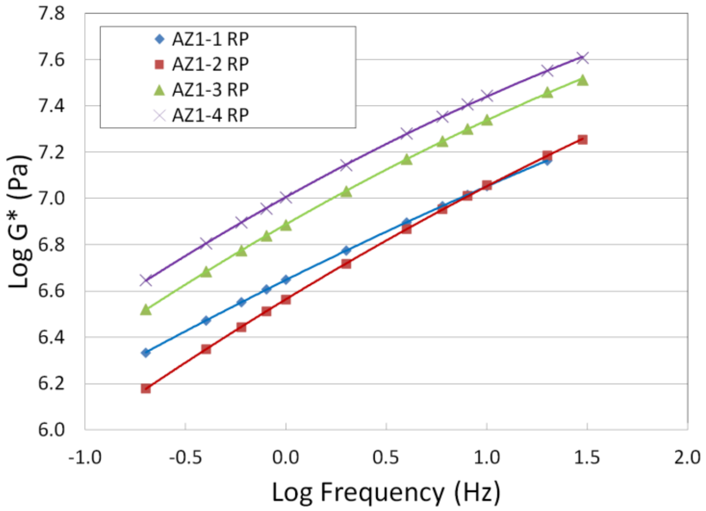

Figure 2. AZ bitumens: frequency sweeps at the intermediate test temperature (28°C).

2.1.3 Perform the Strain Ramp Function

To perform the strain ramp, the strain is increased linearly from 0.1 to 30% over the course of 3,100 cycles of loading at 10Hz for a total test time of 310 sec (0.1% strain per second). Peak shear strain, shear stress, phase angle, and complex shear modulus are recorded every 10 load cycles (1 sec) [TP 101-12-UL].

Strain output for the AZ bitumens is provided in Figure 3a and the effective stress and strain plots are presented in Figure 3b. Figure 3a shows the Malvern Kinexus rheometer used in this study adequately and with good precision performed the required strain ramp function.

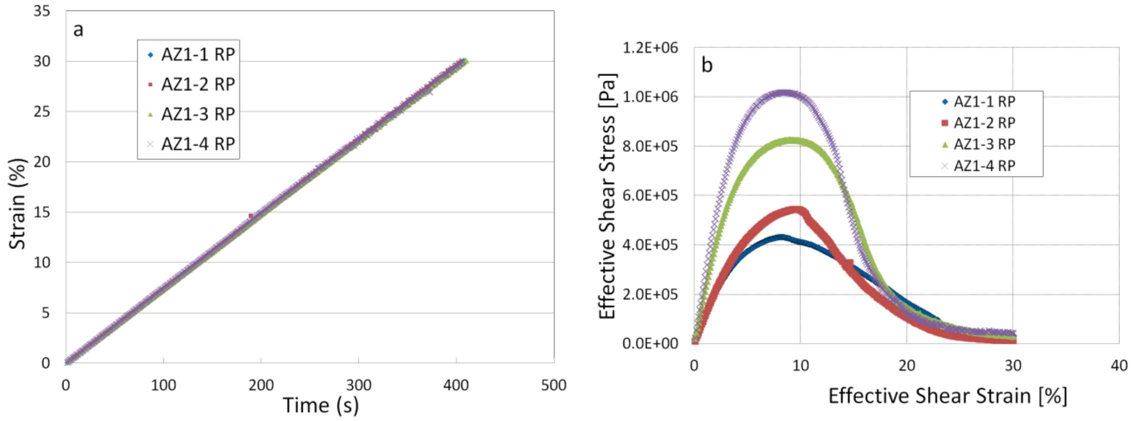

Figure 3 (a): recorded strain, and (b): apparent stress response from the continuous strain sweep.

2.1.4 Analyze the Frequency Sweep and Strain Ramp Results

Details and specific formula for the VECD Damage analysis can be found in the test method TP 101-12-UL.

The first step is to calculate the α parameter from the frequency sweep test data. The storage modulus, G'(ω), is determined by direct conversion of G*(ω) and δ(ω). A best-fit straight line is then applied to a log-log plot G'(ω) versus (ω), and the inverse of the slope of the best fit line is equal to α.

The next step is to calculate the damage accumulation, $D(t)$. Damage accumulation is a function of the applied strain, the normalized loss modulus G", α, and testing time. The normalized loss modulus, G" at each time step is referred to as $C(t)$.

The relationship between $C(t)$ and D(t) is fitted to a power law

$$C(t) = C_0 - C_1(D(t))^{C_2}$$

where C_0 ,C_1 and C_2 are model coefficients. The relationship between $C(t)$ and $D(t)$ for all the bitumens is shown in Figure 4. The relationship is thought to be characteristic of a material's resistance to damage.

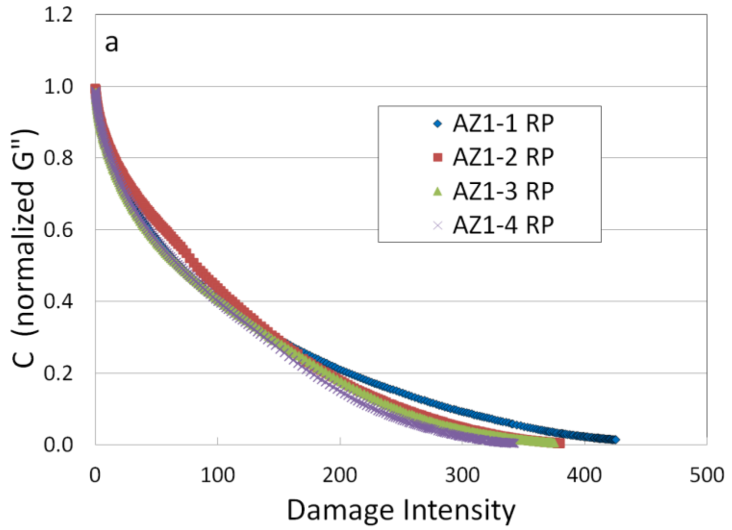

Figure 4. AZ1-1, 2, 3, and 4. Normalized loss modulus, G'' versus damage from LAS testing.

The next step is to use the bitumen fatigue performance model to calculate the number of cycles to failure, N_f. The form of the fatigue model is:

$$N_f = A \times (\text{Amplitude of Applied Load})^{-B}$$

where A is a function of $D(t)$ corresponding to a 35 percent reduction in the initial G", C_1, C_2, and α, and B is solely a function of α.

The N_f results are compared in Figure 5. Strain levels of 2.5 and 5.0% (which are specified by TP 101-12-UL) are considered relatively low, whereas a strain level of about 12% would be considered high. At high strain level, aging typically has a negative effect on longevity. An applied strain of 12% causes the N_f to drop to ~ 30 for all four bitumens.

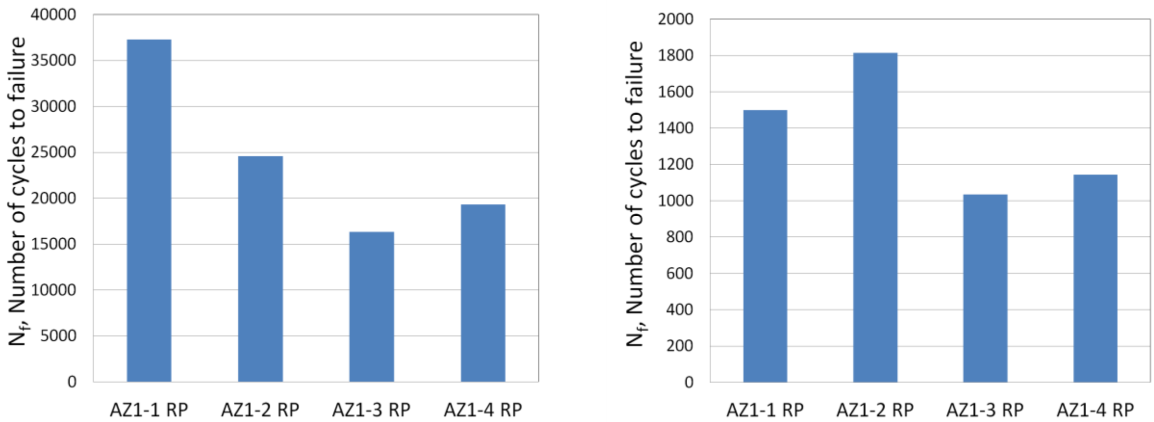

Figure 5. VECD analysis, N_f at 2.5 and 5.0% strain.

2.2 LAOS TEST PROCEDURE AND ANALYSIS

As stated in the introduction, the LAOS test in this study is similar to the LAS test in that it is performed as a strain ramp. In the LAOS test, however, there is no need to run a frequency sweep. Additionally, the strain ramp is run at 1Hz instead of 10Hz in order to prevent potential artifacts due to inertia effects. Because the test is run at 1Hz rather than 10Hz, the strain ramp is run at every 1% strain instead of every 0.1% strain. This keeps the total time of the strain ramp the same as in the LAS test, and it also keeps the amount of real-time data generated at a manageable amount.

The LAOS test attempts to measure the dissipated energy of a sample experiencing cyclic loading at strains outside of the linear viscoelastic region. The real-time torque values are measured by the rheometer and subsequently converted to strain and stress values, which are in turn plotted parametrically as Lissajous curves. The area of a Lissajous curve is equal to the dissipated energy per unit volume during a complete strain cycle. By calculating the dissipated energy of samples this way, calculations of the dynamic moduli are avoided and hence so are the inaccuracies of using only the first harmonic outside the linear viscoelastic region. The dissipated energy calculated from the Lissajous curves is comparable to the dissipated energy from the hysteresis curves often developed from oscillatory mix fatigue tests.

For this study, a Lissajous curve was plotted for each sample from real-time stress-strain data corresponding to the time during the strain ramp when a 10% strain load was being applied. The Lissajous curves for each of the four samples tested in this study are shown superimposed in Figure 6. The areas of the ellipses were numerically integrated using the trapezoid rule to obtain the dissipated energy, in J/m^3, for each sample. The calculated dissipated energies for each sample are shown in Figure 7.

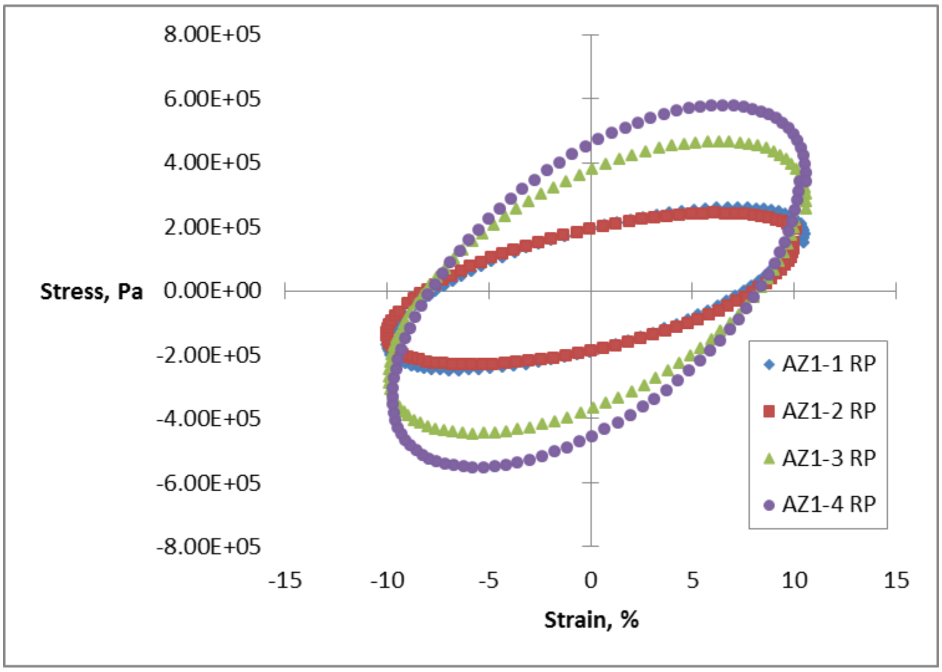

Figure 6. Lissajous curves for each sample at 28C and 10% strain.

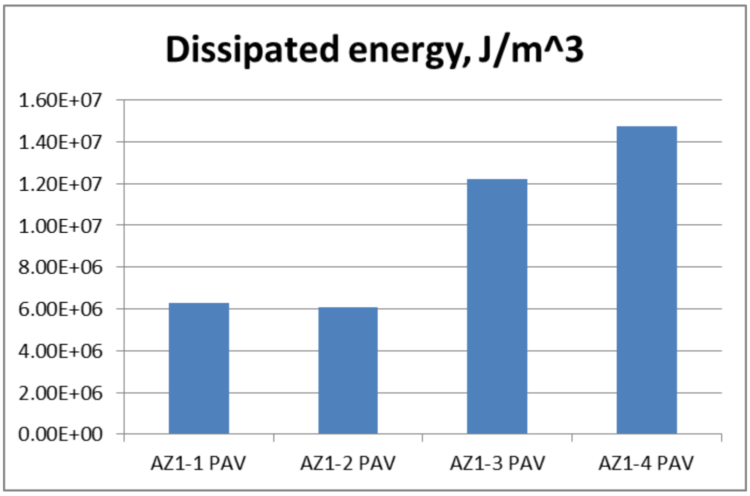

Figure 7. Calculated dissipated energies for samples AZ1-1,2,3, and 4.

2.3 Comparison of LAS and LAOS Parameters to AZ Field Cracking

Meanwhile, in the frame of the field site validation studies carried out during the ARC program, field cracking data were regularly obtained thanks to yearly field surveys from 2001 to 2012. Figures 8 and 9 show the field cracking data after 12 years in 2012 from the AZ test site compared to the results from the LAS and LAOS tests, respectively.

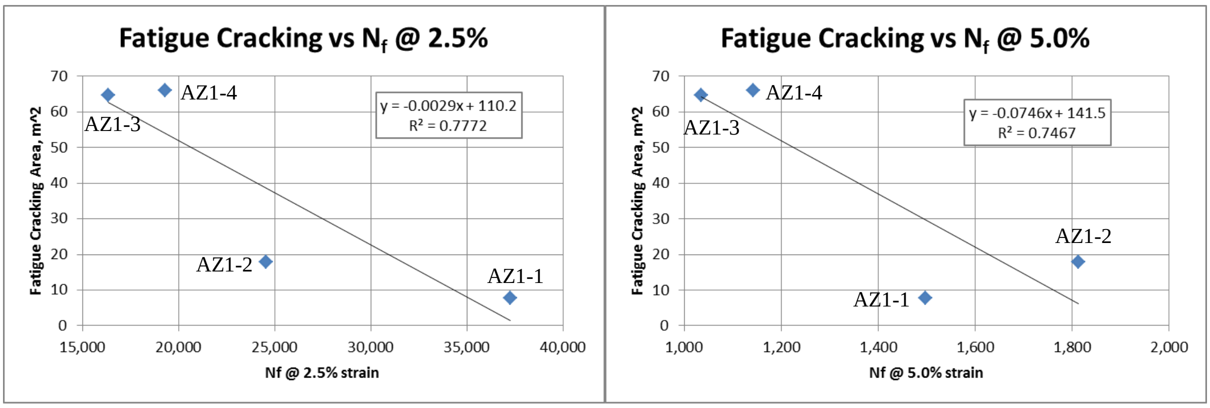

Figure 8. Field fatigue cracking data vs. LAS test N_f values at (a) 2.5% strain and (b) 5.0% strain.

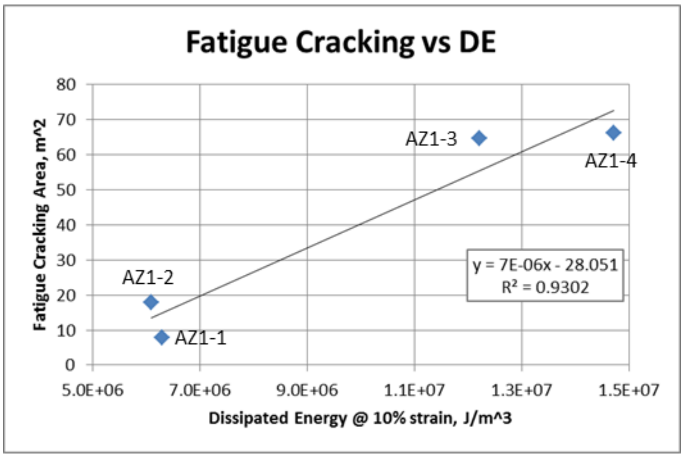

Figure 9. LAOS test dissipated energy (DE) values vs. field fatigue cracking data.

Note that the DE values correlate with the field cracking data better than the N_f values do, showing a higher correlation coefficient above 0.93, but trend in the opposite direction. Whereas increasing N_f values correlate to less fatigue cracking, increasing DE values correlate to more fatigue cracking. This is likely because plastic deformation gives rise to dissipated energy and thus the samples that dissipate more energy are deforming more under load. Or, in other words, the samples that have low dissipated energies are behaving more elastically at the test conditions and are therefore better able to recover from load.

3. CONCLUSIONS AND RECOMMENDATIONS

3.1 Conclusions

- Four asphalt bitumen samples that were used in the wide Asphalt Research Consortium program at the Arizona (AZ) test site were RTFO/PAV aged and tested using the LAS test protocol and a newly designed LAOS test protocol.
- The number of cycles to failure (N_f) from the LAS test results and the dissipated energy (DE) from the LAOS test results were compared to fatigue cracking data measured at the AZ field test site after 12 years. The DE values from the LAOS test correlate more closely to the field cracking data than the N_f values from the LAS test.
- The trend in DE vs fatigue cracking suggests bitumens with high DE values are more prone to fatigue cracking. This may be because these bitumens are deforming plastically instead of elastically at the prescribed test conditions and are therefore unable to recover well from the applied strain.

3.2 Recommendations

- Most importantly, the sample size of future studies should be greatly expanded to include samples used at other test sites around the USA, or elsewhere, as well as differently aged samples (neat, RTFO-only, 2x PAV, etc.).
- Analysis of the linear viscoelastic region limits of samples at multiple temperatures should be performed. This will ensure test conditions elicit similar stress responses from different samples, and may also serve as another basis of fatigue prediction.
- Calculations of the DE at additional strain levels will help identify those most closely related to fatigue cracking.
- Characterization of the shape of the Lissajous curves may aid in determination of the type of response of the sample at the test conditions (elastic/plastic/viscous response).
- Characterization at various iso stiffness temperatures would help to answer true question regarding fatigue: once in the “fatigue domain” for stiffness, what is the actual impact of stiffness and temperature on the damage function?

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