Multiscale Approach for Characterization of Asphaltic Materials Designed in Brazil and Spain

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ABSTRACT: This paper presents multi-scale experimental test protocols to quantify the linear viscoelastic properties of asphaltic materials. The partial results presented are part of an ongoing cooperative research effort between Brazil and Spain, and contains materials from both countries. The evaluation was based on rheological tests conducted with asphalt cements (ACs) and fine aggregate matrices (FAMs) using the dynamic mechanical analyzer (DMA), as well as the dynamic modulus test performed with the global hot mixture asphalt (HMA). The FAM used in the study was designed to represent a proper FAM proportion of the full mixture, and it was tested by applying oscillatory torque under controlled-strain conditions. In general, to reduce costs and time, several researchers have been trying to predict HMA properties using their individual constituent (binder, aggregate, fine aggregate matrices) properties. The authors of the present study intend to suggest the use of individual constituent characterization as an intermediate step aiming time, material and cost savings. Once the ACs and FAMs are properly designed and selected, HMA characterization should be conducted.

KEY WORDS: Asphalt mixture, fine aggregate matrix, asphalt cement, multi-scale approach.

1 INTRODUCTION

Pavement asphalt mixtures are a combination of asphalt binder, aggregates and filler, and can be classified as composite materials. The global properties of such materials depend on the properties of their constituents, their volume fraction and their interaction. The so-called multi-scale modeling has been applied for asphalt mixtures to help understand the role of individual constituents in the performance of the composite (Soares et al. 2003; Souza et al. 2004; Lutif et al. 2010).

Distresses such as fatigue cracking and rutting are related to small scale phenomena, and they are affected by the asphalt content, characteristics of the aggregates such as shape, size, and particle distribution, as well as by the properties of the binder-aggregate interface. A reduction on the asphalt content may reduce the mixture susceptibility for rutting. The corresponding increase on the aggregate volume fraction can increase the mixture susceptibility to cracking. A challenge in mixture design is to find the optimum volume fractions of the constituents in order to provide a material with optimum performance for the considered application.

The aim of the current paper is to present experimental results which are part of an ongoing cooperative research project between Brazil and Spain, consisting of the characterization of asphalt mixture constituents at different scales. Only the viscoelastic properties of binders, fine aggregate matrices, FAMs (asphalt cement plus fine aggregates and filler) and full hot mixtures asphalt, HMAs (FAM plus coarse aggregates) are presented herein. Rutting and fatigue properties will be presented in subsequent papers by the authors.

2 BACKGROUND

HMA characterization has been conducted using two scales: (i) the entire asphalt mixture (coarse and fine aggregates, filler – aggregate smaller than 75μm, and asphalt binder), and (ii) fine aggregate matrix (FAM) (fine aggregate, filler and asphalt binder). Recent work at Texas A&M University proposed an experimental testing procedure that is conducted on the FAM portion of the asphalt mixture, which has a relatively more uniform internal structure compared to the entire HMA (Figure 1).

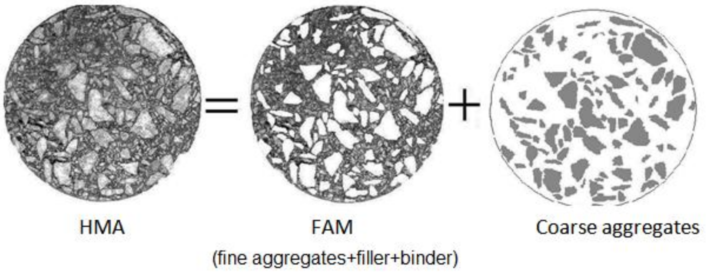

Figure 1: HMA and FAM illustrations.

The FAM represents an important length scale that is intermediate between the HMA and the asphalt cement. The characterization of the fine portion was also motivated by the fact that this part of an HMA influences crack formation and growth (Masad et al. 2006). The effect of the fine portion can be related to the physical properties and volumetrics, which depend on shape properties of the fines, and it can also be of chemical nature, related to the chemical properties of the fines. The interaction at the FAM-coarse aggregates interface is a complex system to evaluate since it depends on both physical and chemical aspects, including the size of the contact surface, which affects the intensity of the binder adsorption at this interface (Souza 2007).

Kim et al. (2003) used the DMA to characterize the fatigue cracking and healing properties for FAMs. These authors conducted controlled-strain tests at 25ºC by applying cyclic loads in a sinusoidal wave form at a frequency of 10Hz. The change in dynamic modulus, pseudo stiffness (relationship between physical stress and pseudo strain) and dissipated strain energy were monitored for tests performed using different strain amplitudes and considering the effect of rest periods. Zollinger (2005) used DMA tests to evaluate the moisture damage susceptibility of eight different types of FAMs. The ratio between the number of cycles to failure under wet and dry conditions was used as an indicator of the mixtures' resistance to moisture damage. Masad et al. (2007) developed an analysis method to unify results from different modes of loading (controlled-strain and controlled-stress). These authors verified the efficacy of their approach using DMA tests and three different types of FAMs. Arambula et al. (2007) evaluated moisture susceptibility of HMA using DMA and a fracture mechanics model. Findings from this study showed similar results (in terms of moisture sensibility) for both the

HMA (characterized using relaxation and uniaxial dynamic tension tests) and the corresponding FAM (characterized using DMA). Caro et al. (2008) used DMA results to characterize fine matrices of four asphalt mixtures and to conduct a probabilistic analysis of fracture caused by moisture damage. Castelo Branco et al. (2008) used a fracture mechanics based crack growth model and DMA tests to evaluate the approach proposed by Masad et al. (2007) for different stress (strain) amplitudes. Vasconcelos et al. (2009) used DMA tests to evaluate the impact of reducing mixing and compaction temperatures on the mechanical behavior of six FAMs designed from six warm mixtures asphalt (WMAs) which used synthetic zeolite.

The time-temperature superposition principle (TTSP) states that data obtained at different temperatures T can be shifted horizontally by a certain value a_t relative to a reference temperature T_{ref}, where T is the test temperature. This process results in aligning the various curves to form a single long-term viscoelastic curve called master curve, either in time or frequency domain (Christensen 1982; Klompen and Govaert 1999; Clyne et al. 2003). There are several different procedures to determine the shift factor. Two well known are the equations by: (i) Arrhenius and (ii) Williams-Landel-Ferry (WLF). The first is one of the most commonly found in the asphalt mixture literature, and it was used in the present study. It is given by:

$$\log a_T = C.\left(\frac{1}{T}-\frac{1}{T_{ref}}\right)=\frac{E_a}{2.303.R}\left(\frac{1}{T}-\frac{1}{T_{ref}}\right) \qquad (1)$$

Where C is the material constant (K), E_a is the activation energy (J/mol), R is the ideal gas constant (8.314J/mol.K), T is the experimental temperature (K), T_{ref} is the reference temperature (K), and 2.303 corresponds to $ln10$. The literature presents E_a values for different asphalt cements ranging from 44kJ/mol to 205kJ/mol. These values impact the C values for different asphalt mixtures. Medani and Huurman (2003) present different values for C, such as 7,680K; 10,920K; and 13,060K. For the asphalt mixtures investigated in the present study, C was considered 10,500K.

3 EXPERIMENTAL INFORMATION

3.1 Materials

As previously mentioned, this investigation is performed with materials from both Brazil and Spain, as part of a cooperative ongoing research effort. From the Brazilian side, two different binders were used: (i) a conventional asphalt cement (AC) produced by Petrobras/Lubnor, classified by penetration as an AC 50/70; (ii) the same binder modified with 4.5% SBS. The Brazilian aggregates are from a granite source typically used in the state of Ceará: coarse aggregates of sizes ¾" and 3/8"; fine aggregates and natural filler from the same granite source. From the Spanish side, an AC 50/70 was also used. The coarse aggregates are siliceous-calcareous mainly from Madrid, and so are the calcareous fine aggregates.

3.2 Procedures

HMA Design

The design of the Brazilian mixtures (HMAs) followed the Superpave methodology using 100 gyrations. These dense-graded mixtures are typically used as pavement surface courses in

Northeast Brazil, mostly with unmodified binders, since there is no heavily loaded traffic. Specimens have 100mm diameter and 150mm height. The Spanish design followed the Marshall methodology, and the mixture investigated is typical for asphalt layers which are still covered by an open mixture placed for friction and drainage purposes. Therefore the objective of the mixtures within pavements in the two countries is not the same. The gradations of the mixtures are presented in Figure 2, which includes not only the aggregate particle distribution of the HMAs, but also of their fine portions, i.e., the FAMs from both Brazil and Spain. The Brazilian gradation is the same regardless of the binder, pure or modified. Table 1 presents design parameters for the mixtures, an average of two specimens.

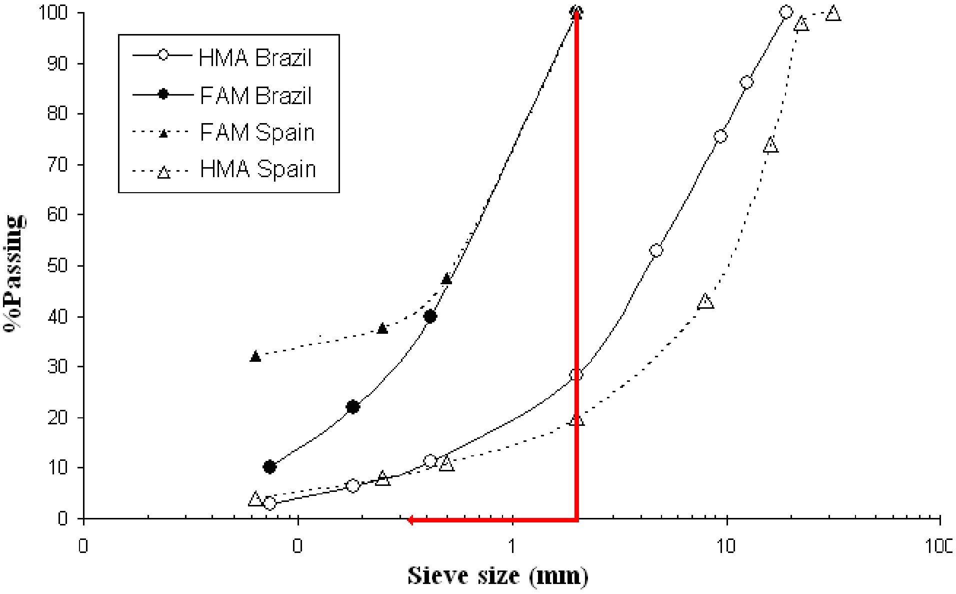

Figure 2: HMA and FAM aggregate gradations.

Table 1: Mixture design parameters

Parameters / Binder	AC 50/70 (Brazil)	AC 50/70 + 4.5% SBS (Brazil)	AC 50/70 (Spain)
Binder (%)	6.0	6.3	3.5
Voids (%)	4.3	3.9	6.7
G_{mm}	2.401	2.397	2.513

FAM Design

FAMs were designed using the methodology proposed by Castelo Branco (2008). The difference between the procedure used during this study to prepare DMA samples from the ones proposed by Kim and Little (2004) and Zollinger (2005) relies on the fact that for the present study the fine aggregates' proportions were similar to their proportions in the HMA. The amount of binder used to prepare FAM samples was the content obtained in the HMA design minus 30% by mass. This simplification was assumed to be reasonable considering that the surface area is more affected by the percent of aggregate passing the smaller sieve sizes and that part of the binder is absorbed by the coarse aggregates (Roberts et al. 1996).

Fine aggregate and binder were combined at mixing temperature, which corresponds to the temperature necessary to take the binder to a viscosity of 160 ±20 centipoise (Jung 1994). Before compaction, each FAM was short term aged in the oven for two hours at mixing temperature. FAM test specimens of 50mm height and 12mm diameter were prepared (Figure 3). Approximately 10 FAM samples are obtained by coring a 100mm diameter and 90mm high specimen compacted using a Superpave gyratory compactor (SGC). To obtain the desired height (50mm) for the FAM sample and also to assure uniformity, the ends of the 100mm SGC sample were sawed off before coring (Figure 3b). More details about the FAM preparation procedure can be found in Castelo Branco (2008).

(a) SGC source specimen

(b) Process of sawing SGC specimen

(c) Process of coring FAM specimens

(d) FAM final specimens

(e) Testing FAM specimens using the DMA

Figure 3: Production and testing of FAM specimens.

Table 2: FAM design parameters

Parameters / Binder	AC 50/70 (Brazil)	AC 50/70 + 4.5% SBS (Brazil)	AC 50/70 (Spain)
Binder (%)	11.7	12.2	9.4
Voids (%)	0.9	0.8	1.5
G_{mm}	2.157	2.159	2.307

Laboratory Tests

Dynamic modulus tests were performed for the HMAs using a *Universal Testing Machine 25* (UTM 25), and following AASHTO TP 62-03. A semi-sinusoidal compression axial stress is applied on the specimen for different frequencies and temperatures. For the present study, five temperatures were used: -10; 4.4; 21.1; 37.8; 54.4°C, and six frequencies: 25; 10; 5; 1; 0.5; 0.1Hz.

FAMs and ACs were tested in a dynamic mechanical analyzer (DMA), model TA AR

2000® (Figure 3). The dynamic modulus is determined after submitting the samples to oscillatory shear stresses. For the ACs (pure and modified), frequency sweep tests under a controlled-stress of 120Pa were performed. The tests were performed in two different temperature ranges: -10 to 40°C, and 40 to 85°C. For the two ranges, frequencies varied from 0.01 to 1Hz. For the FAMs, the 12mm diameter by 50mm height specimens were used. Temperatures were -10; 4.4; 21.1; 37.8 and 54.4°C, and the frequency ranged from 0.01 to 15Hz for each temperature. The test was performed under controlled-strain with a strain amplitude of 10^{-5}με.

4 RESULTS

4.1 Frequency Sweep Tests for the ACs

The results for the three ACs investigated are presented in Figure 4. A reference temperature of 20°C was used for constructing the master curves. For the Brazilian binder one can note that the SBS increases the stiffness particularly for low frequencies, which is important for the material resistance to rutting. At high frequencies the effect of the modifier is not observed. The Spanish binder behavior was very similar to the conventional Brazilian AC 50/70, for low and high frequencies. As the curves presented are continuous, the TTSP holds for the test conditions, implying that the materials are thermo-rheological simple for such conditions.

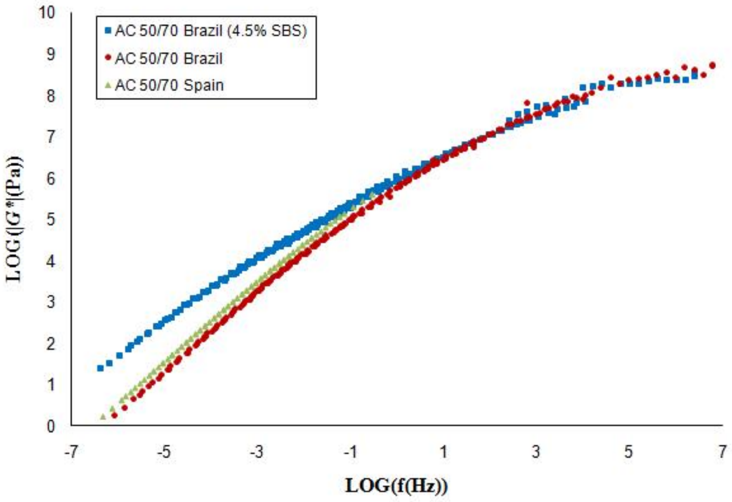

Figure 4: Master curves of the investigated asphalt cements (ACs), reference temperature of 20°C.

4.2 Frequency Sweep Tests for the FAMs

For the FAM frequency sweep tests, two temperatures were added: 10 and 25ºC. Because of limitations of the DMA related to its torque capability, tests were not conducted in the FAMs at low temperatures (-10 and 4.4ºC). A reference temperature of 21.1°C was used for constructing the master curves (Figure 5).

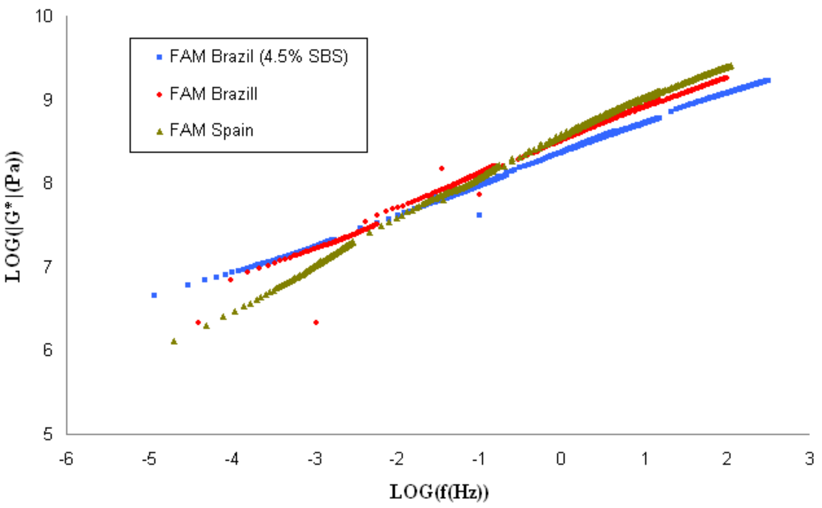

Figure 5: Master curves of the investigated FAMs, reference temperature of 21.1°C.

Brazilian FAM curves are very similar even for the FAM designed using the modified AC. Master curves in Figure 5 do not have a S-shape, which is very common for those materials. The authors suspect that this happened due to the lack of data for low temperatures. The Spanish FAM presented higher stiffness if compared to the Brazilian FAMs for high frequencies, and lower stiffness for low frequencies. This was expected due to the lower asphalt content of this mix (Table 2).

4.3 Dynamic Modulus – HMAs

The stiffness results for the three HMAs investigated are presented in Figure 6. The shift factors (a_t) were obtained using the Arrhenius equation, adopting the same constant C (10,500K) for the different mixtures. As expected, the dynamic modulus increases with an increase in frequency.

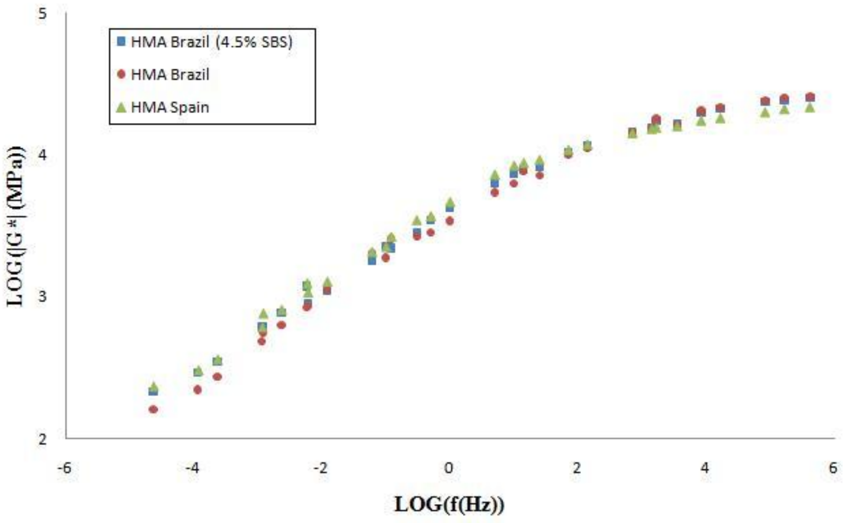

Figure 6: Master curves of the investigated HMAs, reference temperature of 21.1°C.

As with the master curves of the ACs and FAMs, the HMA master curves are continuous.

Therefore, the TTSP holds for the test conditions, implying that the materials are thermo-rheological simple for such conditions. Although the HMAs are composed by different materials (sources and quantities) and were designed by different methodologies (Superpave and Marshall), the dynamic modulus behavior for different frequencies and temperatures were similar for the three HMAs. The differences in stiffness on both scales (FAM and HMA – Figures 5 and 6, respectively) can be attributed to the coarse aggregates stiffness. Aragão et al. (2009) reported aggregate (gravel and limestone) Young's modulus, measured by nano-indentation technique, in the order of 50.8GPa.

A multi-scale characterization can be justified especially when one intends to predict mixture behavior. In one hand, there is a great risk when trying to access mixture performance based only on binder information. On the other hand, the fact that the HMA is commonly a highly heterogeneous material contributes to the limitation of its characterization with respect to fatigue damage and permanent deformation. Heterogeneity in itself limits one obtaining material properties, with tests presenting great coefficients of variation. According to Dai et al. (2006), HMA macro load-carrying behavior depends on many micro-phenomena that occur at aggregate and FAM level. FAM properties that should be observed are: (i) volume percentage, (ii) elastic/viscoelastic moduli, (iii) damage and time-dependent responses, (iv) age hardening, (v) microcracking, and (vi) debonding from coarse aggregates.

To reduce costs and time, several researchers have been trying to predict HMA properties using their individual constituents (binder, aggregates, fine matrices) properties. Masad et al. (2006) developed a new approach to predict fatigue damage that should be applied first for FAMs. These authors believe that full mixture results are highly influenced by the complexity and heterogeneity of the internal structure, which could hinder the efforts to link properties of mixture constituents to fatigue resistance. Arambula (2007) developed a fracture mechanics approach to access the influence of air void structure on the moisture susceptibility of asphalt mixtures. This model uses adhesive bond surface energy, viscoelastic properties, dissipated energy due to damage and tensile strength. The validity and applicability of the crack growth model were studied using asphalt mixtures and the correspondent FAM using DMA tests. For the three mixtures analyzed, both FAM and HMA results ranked in the same way with respect to moisture damage resistance. Aragão et al. (2009) proposed a finite element micromechanics model-based to predict the dynamic modulus of the asphalt mixture using linear viscoelastic material properties of each mixture constituent (asphalt matrices and aggregates). To characterize FAM, dynamic frequency sweep tests for the material linear region were conducted. Model results using fine matrices and aggregates properties were compared to dynamic modulus tests conducted in two different dense-graded HMAs. The proposed model presented good agreement with experiments and significant savings in cost and time. For multi-scale FEM modeling of asphalt mixtures, there is still need for further research on how to determine the characteristics of the FAM-coarse aggregate interface. Freitas (2007) has developed an attempt for experimentally determining the constitutive behavior of such interface.

The present study did not make any attempt of computational modeling. It has focused on presenting experimental results, suggesting that the characterization of individual mixture constituents can be an intermediate step, and important for time, material and cost savings. Once ACs and FAMs are properly designed and selected, reduced and more efficient HMA experiments should be conducted.

5 CONCLUSIONS

The methodology used to design FAM needs to be further improved in order to guarantee a representative sample of the material that interacts with the coarse aggregates in the overall HMA. Questions still remain with respect to the proper binder content that needs to be used in such fine mixtures. FAM testing with DMA was found to work properly, but difficulties were encountered to conduct the test at low temperatures.

The time temperature superposition principle (TTSP) holds for all three scales investigated, indicating that the materials can be treated as thermo-rheological simple. The Arrhenius equation fitted well the experimental data considering the same constant C regardless of the material.

Similar results (master curves) were found for the three HMAs analyzed, even though a modified binder was used in one of the mixtures and has presented greater stiffness at low frequencies (higher temperatures). In other words, such binder characteristic was not passed on for the mixtures with coarse aggregates.

Further testing will be performed (including fatigue and permanent deformation) in order to determine whether global mixture characteristics can be inferred from testing within other scales (ACs, and especially FAMs). Such smaller-scale testing requires much less material and time, and has a more homogeneous structure. Therefore, they can represent a great gain in efficiency if it is proved that they can provide similar trends to the global testing, with respect to the most adequate constituent proportioning in mixture design.

REFERENCES

Aragão, F.T.S., Kim, Y., Lee, J., Soares, J.B., 2009. *A Micromechanical Finite Element Model for Predicting the Dynamic Modulus of Heterogeneous and Rate-Dependent Asphalt Concrete Mixtures*. In Proceedings of the IV Simpósio Internacional de Avaliação de Pavimentos e Projetos de Reforço. Fortaleza, CE, Brazil.

Arambula, E., 2007. *Influence of Fundamental Material Properties and Air Voids Structure on Moisture Damage of Asphalt Mixes*. Ph.D. Dissertation, Texas A&M University, College Station, TX, USA.

Arambula, E., Masad, E., Epps Martin, A., 2007. *Moisture Susceptibility of Asphalt Mixtures with Known Field Performance Using Dynamic Analysis and a Crack Growth Model. In* Transportation Research Record: Journal of the Transportation Research Board. N^o 2001, TRB, National Research Council, Washington, D.C., 20-28.

AASHTO TP 62-03, 2005. *Standard Method of Test for Determining Dynamic Modulus of Hot-Mix Asphalt Concrete Mixtures*, American Association of State Highway and Transportation Officials, Washington, D.C.

Caro, S., Masad, E., Airey, G., Bhasin, A., Little, D., 2008. *Probabilistic Analysis of Fracture in Asphalt Mixtures Caused by Moisture Damage*. In Transportation Research Record: Journal of the Transportation Research Board. N^o 2057, TRB, National Research Council, Washington, D.C., 28-36.

Castelo Branco, V.T.F., 2008. *A Unified Method for the Analysis of Nonlinear Viscoelasticity and Fatigue Cracking of Asphalt Mixtures Using the Dynamic Mechanical Analyzer*. Ph.D Dissertation, Texas A&M University, College Station, TX, USA.

Castelo Branco, V.T.F., Masad, E., Bhasin, A. Little, D.N., 2008. *Fatigue Analysis of Asphalt Mixtures Independent of Mode of Loading*. In Transportation Research Record: Journal of the Transportation Research Board. N^o 2057, TRB, National Research Council, Washington, D.C., 149-156.

Christensen, R. M., 1982. *Theory of Viscoelasticity*. Academic Press, New York, USA.

Clyne, T. R., Li, X., Marasteanu, M. O., Skok, E. L., 2003. *Dynamic and Resilient Modulus of Mn/DOT Asphalt Mixtures*. Technical Report, Department of Civil Engineering, University of Minnesota.

Dai, Q., Sadd, M.H., You, Z., 2006. *A Micromechanical Finite Element Model for Linear and Damage-Coupled Viscoelastic Behavior of Asphalt Mixture*. International Journal for Numerical and Analytical Methods in Geomechanics, 30, 1135-1158.

Freitas, F.A.C., 2007. *A Theoretical and Experimental Technique to Measure Fracture Properties in Viscoelastic Solids*. Ph.D. Dissertation, University of Nebraska, Lincoln, NE USA.

Jung, D.H., 1994. *Low-Temperature Cracking Binder Validation. Strategic Highway Research Program* (SHRP), SHRP-A-399, National Research Council, Washington, D.C.

Kim, Y.R., Little, D.N., Lytton, R.L., 2003. *Fatigue and Healing Characterization of Asphalt Mixtures*. Journal of Materials in Civil Engineering, Vol. 15, ASCE, 75-83.

Kim, Y.R., Little, D.N., 2004. *Linear Viscoelastic Analysis of Asphalt Mastics*. Journal of Materials in Civil Engineering, ASCE, 122-132.

Klompen, E.T.J., Govaert, L.E., 1999. *Nonlinear Viscoelastic Behaviour of Thermorhologically Complex Materials*. Mechanics of Time-Dependent Materials, Vol. 3 (1), 49-69.

Lutif, J.E.S., Souza, F.V., Kim, Y., Soares, J.B., Allen, D.H., 2010. *Multiscale Approach to Predict the Mechanical Behavior of Asphalt Mixtures*. Paper Accepted for Presentation in the Transportation Research Record: Journal of the Transportation Research Board.

Masad, E., Castelo Branco, V.T.F., Little, D.N., Lytton, R.L., 2006. *An Improved Method for the Dynamic Mechanical Analysis of Fatigue Failure of Sand Asphalt Mixtures*. Publication FHWA-473630. FHWA, U.S. Department of Transportation.

Masad, E., Castelo Branco, V.T.F., Little, D.N., Lytton, R., 2007. *A Unified Method for the Analysis of Controlled-Strain and Controlled-Stress Fatigue Testing*. International Journal of Pavement Engineering, Vol. 9, 233-246.

Medani, T. O., Huurman, M., 2003. *Constructing the Stiffness Master Curves for Asphaltic Mixes*. ISSN 0169-9288.

Roberts, F.L., Kandhal, P.S., Brown, R.E., Lee, D., Kennedy, T.W., 1996. *Hot Mix Asphalt Materials, Mixture Design, and Construction*. NAPA.

Soares, J. B., Freitas, F. A., Allen, D. H., 2003. *Crack Modeling of Asphaltic Mixtures Considering Heterogeneity of the Material. In* Transportation Research Record: Journal of the Transportation Research Board. N^o 1832, TRB, National Research Council, Washington, D.C., 113-120.

Souza, P.C., 2007. *Métodos para a Sistematização da Produção e Ensaio de Corpos de Prova de Mástique*. Federal University of Ceará, Fortaleza, CE.

Souza, F. V., Soares, J. B., Allen, D. H., Evangelista, F., 2004. *Model for Predicting Damage Evolution in Heterogeneous Viscoelastic Asphaltic Mixtures. In* Transportation Research Record: Journal of the Transportation Research Board. N^o 1891, TRB, National Research Council, Washington, D.C., 131-139.

Vasconcelos, K.L., Bhasin, A., Little, D.N., 2009. *Influence of Reduced Production Temperatures on the Adhesive Properties of Aggregates and Laboratory Performance of Fine Aggregate – Asphalt Mixtures*. International Journal of Road Materials and Pavement Design (In Press).

Zollinger, C.J., 2005. *Application of Surface Energy Measurements to Evaluate Moisture Susceptibility of Asphalt and Aggregates*. M.S. Thesis, Texas A&M University, College Station, TX, USA.