

Biasing effects (non-linearity, self-heating, thixotropy) occurring during fatigue tests on bituminous mixtures

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ABSTRACT

The study presented in the paper focuses on the evaluation of biasing effects occurring during laboratory fatigue tests on bituminous mixtures. The impact of biasing effects on complex modulus (norm and phase angle) values obtained during tests is estimated.

Six mixtures were produced with three distinct base bitumens, including both modified and unmodified binders, and with varying contents of Reclaimed Asphalt Pavement (RAP), from 0% to 40%. Strain-controlled tension/compression tests were conducted on cylindrical samples according to a procedure specifically developed at the ENTPE, called ALFABET (Advanced Laboratory Fatigue And Biasing Effects Test). The test procedure is composed of an initial series of short complex modulus tests at varying temperatures and strain amplitudes, followed by five partial fatigue tests. After each fatigue period, a 24 hour rest period is observed, during which short complex modulus tests are performed in order to monitor recovery of mechanical properties. Surface and internal temperatures of samples are constantly monitored throughout the test.

Biasing effects were isolated and quantitatively estimated. The influence of non-linearity, self-heating and thixotropy on norm of complex modulus and phase angle of bituminous mixtures was evaluated, both in absolute and relative terms, with respect to initial values obtained in undamaged conditions.

Keywords: Asphalt, Durability, Healing, Mechanical Properties, Testing

1. INTRODUCTION

Extensive research work is performed on fatigue of bituminous mixtures ([1], [2], [3] among others). However, fatigue behavior of asphalts is not yet fully understood. Results of laboratory investigations do not always correlate with performances of real road pavement structures ([4], [5] among others). The lack of scientific knowledge fatigue behavior is an obstacle for industrial practice.

Laboratory procedures generally performed to test fatigue behavior consists of repetitions of cyclic loading applications until failure. However, road sections are subjected to discontinuous loading due to traffic. This difference has a profound effect on material performances. The absence of rest periods during tests causes the occurrence of so-called "biasing effects", which affect test results and, therefore, the understanding of material behavior ([1], [6], [7], [8]). The most important biasing effects are non-linearity, self-heating and thixotropy (the latest is still a hypothesis).

Recently, a new procedure, called ALFABET (Advanced Laboratory Fatigue And Biasing Effects Test) has been developed to quantify these biasing effects, therefore isolating proper fatigue occurrence ([8]). The objective of this paper is to present further development to the analysis procedure of ALFABET results, in particular, regarding the estimation of complex modulus during rest periods.

2. TESTED MATERIALS

Six distinct asphalt mixtures were produced and tested for the study (Table 1). The same aggregate nature (limestone, from Haut-Lieu quarry, France) and the same 14 mm continuous grading curve (Figure 1) were used for all mixtures. Four different bitumens were used: a 35/50 pen grade straight run bitumen (pen = 34), the same binder modified with 2.5% SBS (pen = 30), an air-rectified 35/50 B ("multigrade") pen grade binder (pen = 37) and a soft paving grade binder modified with a high SBS content, cross-linked ("Orthoprène[®]", pen = 56). Penetration tests were performed on all binders according to EN 1426 norm. As indicated in Table 1, four asphalts were produced with 20% RAP material by weight of the total mixture without fresh binder. One mix contain 40% of the same RAP. The RAP used for mix production was issued from a unique lot. Its binder was extracted according to EN 12697-3 specification in order to measure binder content and perform penetration tests. A 5.22% binder content (0.22% standard deviation) was obtained by performing 36 extractions on 18 samples (2 extractions per sample). Penetration tests were performed on two samples, from two extractions. Obtained values were 9 and 15 dmm, respectively. All of the six asphalts tested were produced with a total 5.35% binder content by weight of the final mixture. Production of mixtures was performed following EN 12697-35+A1. All materials were compacted using a LCPC-type slab compactor (following EN 12697-33+A1). The cylindrical samples (150 mm high, 75 mm diameter) were cored from slabs. Their void content (Table 1) was checked before performing tests. One sample was tested for every material, except for mixture 35/50 + 20% RAP (two samples).

Table 1: Asphalt mixtures tested

Mixture	Fresh binder	RAP content	Void content
35/50	35/50	0%	2.7%
35/50 + 20% RAP	35/50	20%	4.0% (sample A)
			5.3% (sample B)
35/50 + 40% RAP	35/50	40%	3.4%
35/50 + 2.5% SBS + 20% RAP	35/50 + 2.5% SBS	20%	5.4%
35/50 B + 20% RAP	35/50 B	20%	3.7%
Orthoprène [®] + 20% RAP	Orthoprène [®]	20%	4.0%

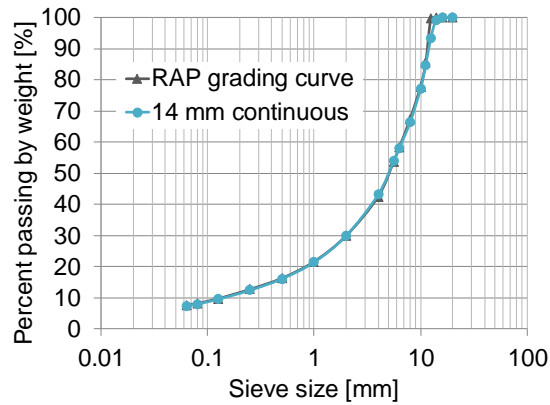


Figure 1: Continuous 14 mm grading curve used for all mixtures (blue) and RAP grading curve (grey)

3. EXPERIMENTAL PROCEDURE: ALFABET TEST

The ALFABET test procedure is summarized in the present paper. For further details, the reader can refer to a previous article ([8]) where it was thoroughly described.

Samples are loaded in tension/compression configuration. Cyclic sinusoidal loading is applied by means of a hydraulic press in strain-control mode. Axial strain is monitored with three extensometers placed at 120° from each other in the central part of the sample (Figure 2). The average value of the three extensometer is retained for the axial strain. Axial stress is obtained with a load cell. Temperature of the sample is constantly measured throughout the whole test. Surface temperature is obtained with a probe applied on the surface. Internal temperature is measured with a 1 mm diameter thermocouple, which is inserted at a 30 mm depth in a 1.7 mm diameter radial hole drilled in the sample. After the insertion of the thermocouple, the hole is filled with bitumen, in order to ensure material continuity.

Test temperatures were maintained by means of a thermal chamber. However, the temperature regulation system of the chamber uses an independent thermocouple, therefore a slight difference between chamber and sample temperatures (approximately from 0.5°C to 0.7°C) is observed after sample conditioning. The effective sample temperature is always considered for the analysis.

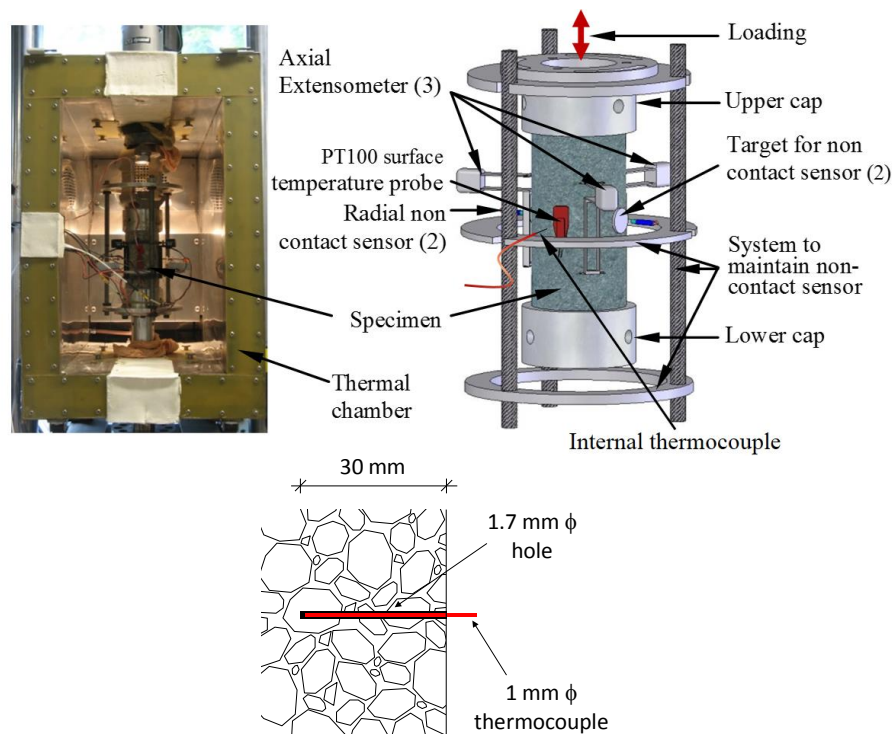


Figure 2: (top, left) Tension-compression test apparatus; (top, right) Detailed scheme of sample and sensors (ENTPE laboratory, Vaulx-en-Velin, France); (bottom) Scheme of internal thermocouple placement (from [8])

The ALFABET test procedure is composed of two parts. In the first part, a series of Complex Modulus Tests (CMT) are performed in order to investigate Linear ViscoElastic (LVE) behavior of asphalt mixtures in undamaged conditions (a scheme of this part of the procedure is shown in Figure 3). For this reason, for each CMT, 200 loading cycles are applied. Four different temperatures (8°C, 10°C, 12°C and 14°C) and four different strain amplitudes (50 $\mu\text{m/m}$, 75 $\mu\text{m/m}$, 100 $\mu\text{m/m}$ and 110 $\mu\text{m/m}$) are used, close to values used for fatigue tests (10°C, 100 $\mu\text{m/m}$). Frequency is always fixed at 10 Hz. A six-hour conditioning period is imposed for every temperature. A 900s rest period is imposed between two consecutive tests performed at the same temperature. The hydraulic press needs approximately between 40 and 50 cycles before the imposed strain amplitude is reached. Data acquired during these cycles were used in the analysis (details in Section 4).

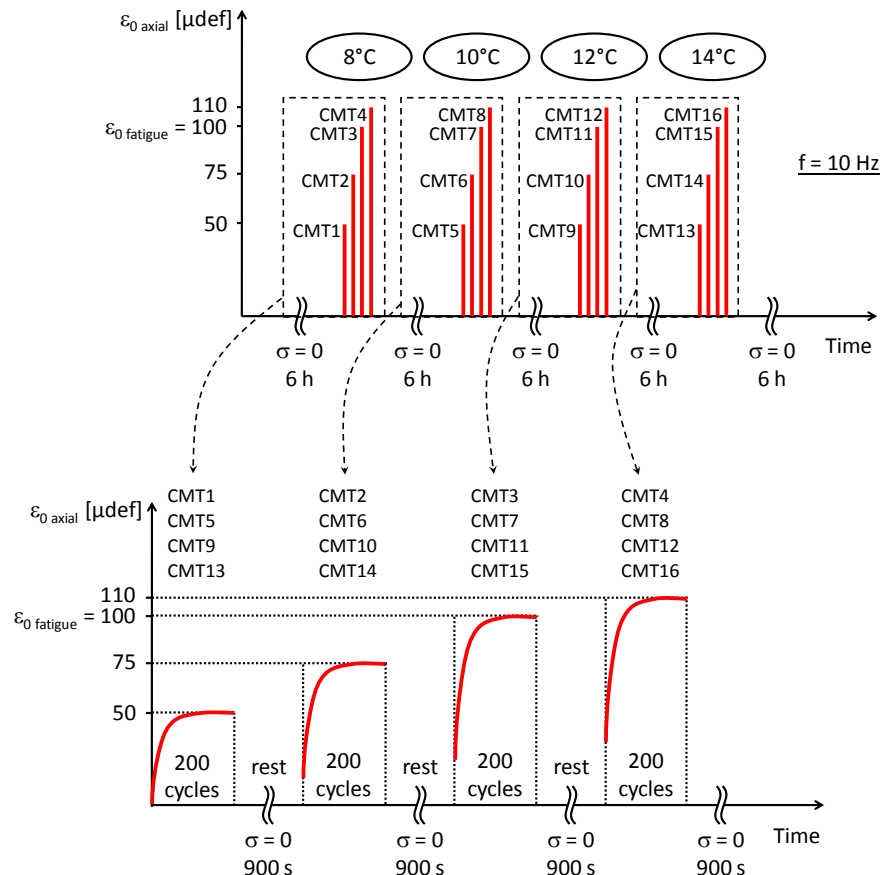


Figure 3: Scheme of the procedure of the first part of ALFABET test, consisting of a series of Complex Modulus Tests (CMT) at different temperatures and strain amplitudes (from [8])

The second part of the ALFABET test consists of a series of Partial Fatigue Tests (PFT). This part is performed immediately after all CMTs on the same sample. A scheme of this part of the ALFABET test is shown in Figure 4. The objective is to quantify biasing effects occurring during fatigue tests. In order to do so, a variation of complex modulus is produced by applying 100 000 cycles of 100 $\mu\text{m/m}$ amplitude during each PFTs. Immediately after each PFT, a 24 hour rest period is imposed, during which short complex modulus tests (of 100 cycles each) are performed at regular intervals (after 10, 20 and 30 minutes and 1, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 22 and 24 hours) to monitor complex modulus recovery. Five consecutive lags are performed, each composed by a PFT and its rest period. For each The recovered part of the total variation of LVE parameters is attributed to various biasing effects, while the unrecovered part is considered to be representative of the real fatigue damage (however, it should be noted that different outcome might result from longer durations of rest periods). The temperature of the thermal chamber is maintained at 10°C during the entire duration of this second part. All loading cycles (both for PFTs and complex modulus tests during rest periods) are performed at 10 Hz.

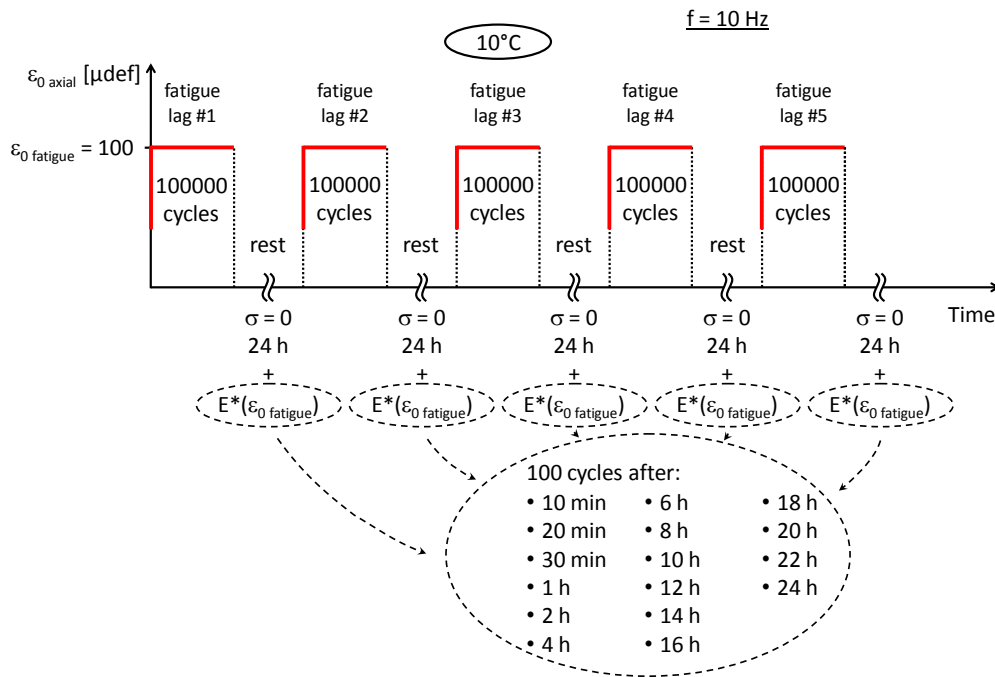


Figure 4: Scheme of the procedure of the second part of ALFABET test, consisting of five fatigue lags, each composed of a Partial Fatigue Test (PFT) and its rest period (from [8])

The whole ALFABET test procedure has a non negligible duration, approximately equal to 7 days. For this reason, the experimental campaign performed for this study included six asphalt mixtures and only one sample was used for each material, except for mixture 35/50 + 20% RAP, for which two samples were tested.

4. RESULTS AND ANALYSIS

In this paper, only test results and analysis regarding $|E^*|$ are described. As already shown in [8], phase angle φ can also be used to quantify biasing effects during fatigue tests. The new analysis procedure performed on φ data confirms the observations obtained for $|E^*|$ (described in the following). However, because of length limitations due to the nature of the article, these results are not reported.

4.1 Complex Modulus Tests (CMTs)

Figure 5 shows values of norm of complex modulus obtained for mixture 35/50 + 40% RAP against corresponding strain level. The first six cycles of each CMT were not taken into account. As strain increases, during the first part of every CMT (approximately 40 or 50 cycles), norm of complex modulus decreases linearly (approximately between -5 and -6 MPa·m/μm). Slightly different linear trends could be observed at different temperatures. No LVE limit could be observed, for strain amplitudes as low as 30 μm/m. Once the imposed strain amplitude reaches a constant value, repeated cycling solicitation causes $|E^*|$ to slightly decrease. In order to quantify the variation rates of $|E^*|$ with strain amplitude, only data obtained during cycles 7-32 of each CMT (at varying strain amplitudes) were fitted with linear regression equations. Thus, non-linearity envelopes were obtained for $|E^*|$ as a function of strain amplitude, at different temperatures.

Non-linearity envelopes were used to estimate $|E^*|$ values at 50 μm/m, 75 μm/m, 100 μm/m and 110 μm/m, for each of the considered temperatures. These values were then plotted against corresponding temperature (Figure 6 shows an example for mixture 35/50 + 40% RAP). The Time-Temperature Superposition Principle ([9]) could not be applied, since all tests were performed at 10 Hz. However, within the limited temperature domain used, linear regressions could successfully be fitted to $|E^*|$ values obtained from non-linearity envelopes. Equation (1) could then be used to estimate $|E^*|$ variations with temperature T (in °C), depending on strain level ε :

$$|E^*|(\varepsilon, T) = b_E(\varepsilon)T + |E^*_{0^\circ\text{C}}|(\varepsilon) \quad (1)$$

where b_E is a coefficient describing $|E^*|$ variation with temperature and $|E^*_{0^\circ\text{C}}|$ is the theoretical extrapolated value of $|E^*|$ at 0°C. Coefficient b_E is used in the analysis of PFT results (Section 4.2).

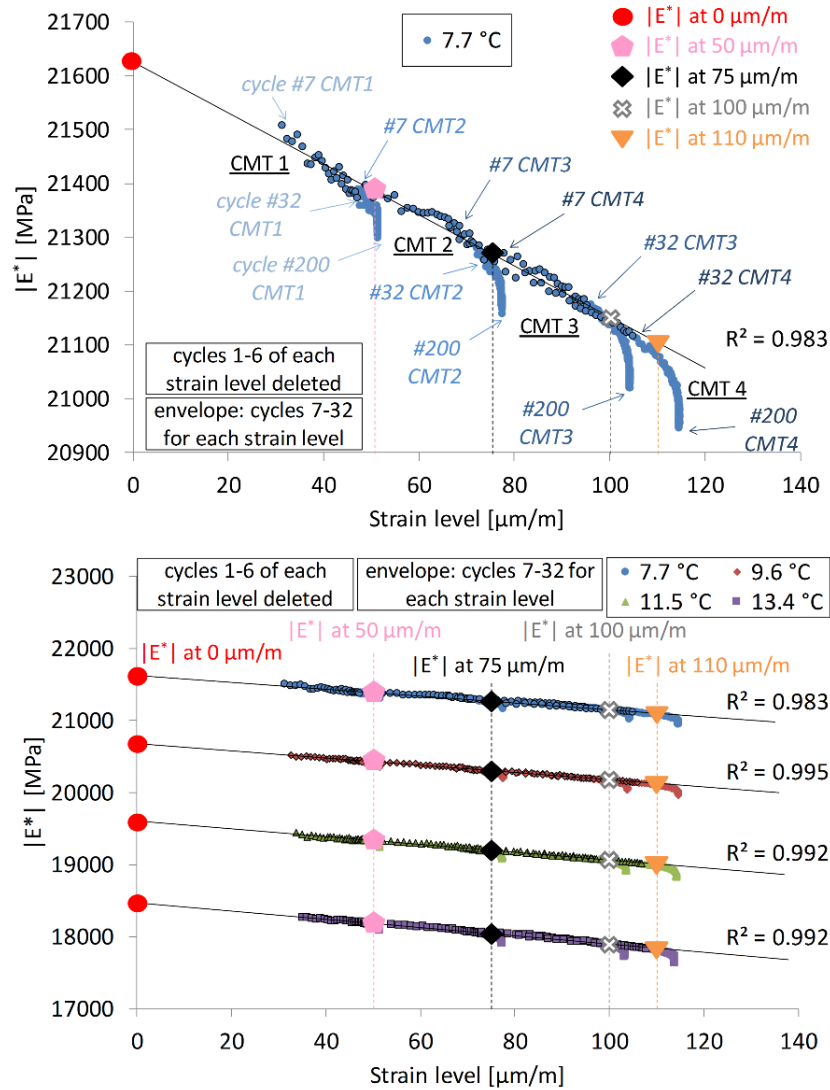


Figure 5: $|E^*|$ data obtained for mixture 35/50 + 40% RAP during CMTs (Complex Modulus Tests): (top) results obtained at 7.7°C at different strain levels; (bottom) non-linearity envelopes at different temperatures (from [8])

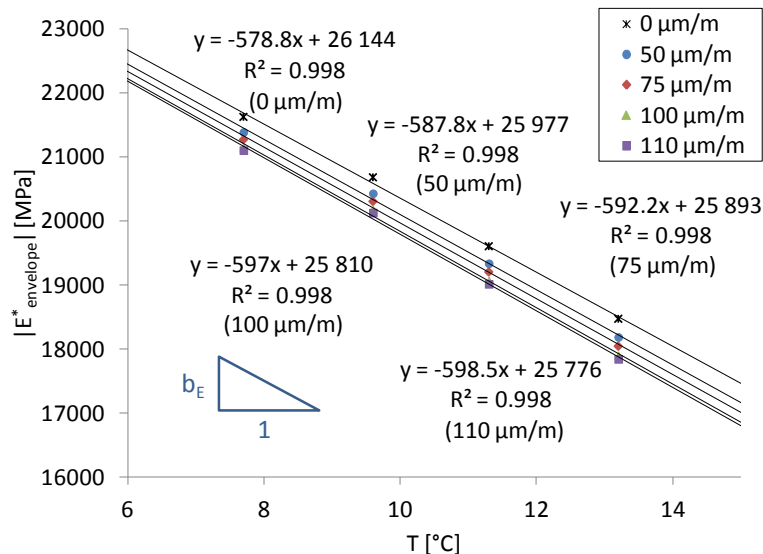


Figure 6: $|E^*|$ data obtained for mixture 35/50 + 40% RAP during CMTs (Complex Modulus Tests): $|E^*|$ values estimated with non-linearity envelopes (Figure 5) plotted against corresponding temperature (from [8])

4.2 Partial Fatigue Tests (PFTs)

Figure 7 shows an example of results obtained during PFTs for mixture 35/50 B + 40% RAP. At the beginning of each cyclic loading period (both during fatigue lags and recovery), applied strain level increases gradually before reaching the targeted value. For each i th fatigue lag, $|E^*|$ values obtained during cycles 7-32 were taken into account to calculate a non-linearity envelope. The regression equation of each envelope was then used to estimate $|E^*|$ values corresponding to 100 $\mu\text{m}/\text{m}$ ($|E^*_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}|$, indicated by red stars in Figure 7) and, virtually, 0 $\mu\text{m}/\text{m}$ ($|E^*_{0\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}|$). T_0 is the internal temperature of the sample measured at temperature equilibrium at the beginning of each fatigue lag (it does not show significant variations among different lags). It can be observed that although $|E^*|$ shows a significant reduction during each fatigue lag, its variation is almost completely recovered during rest periods.

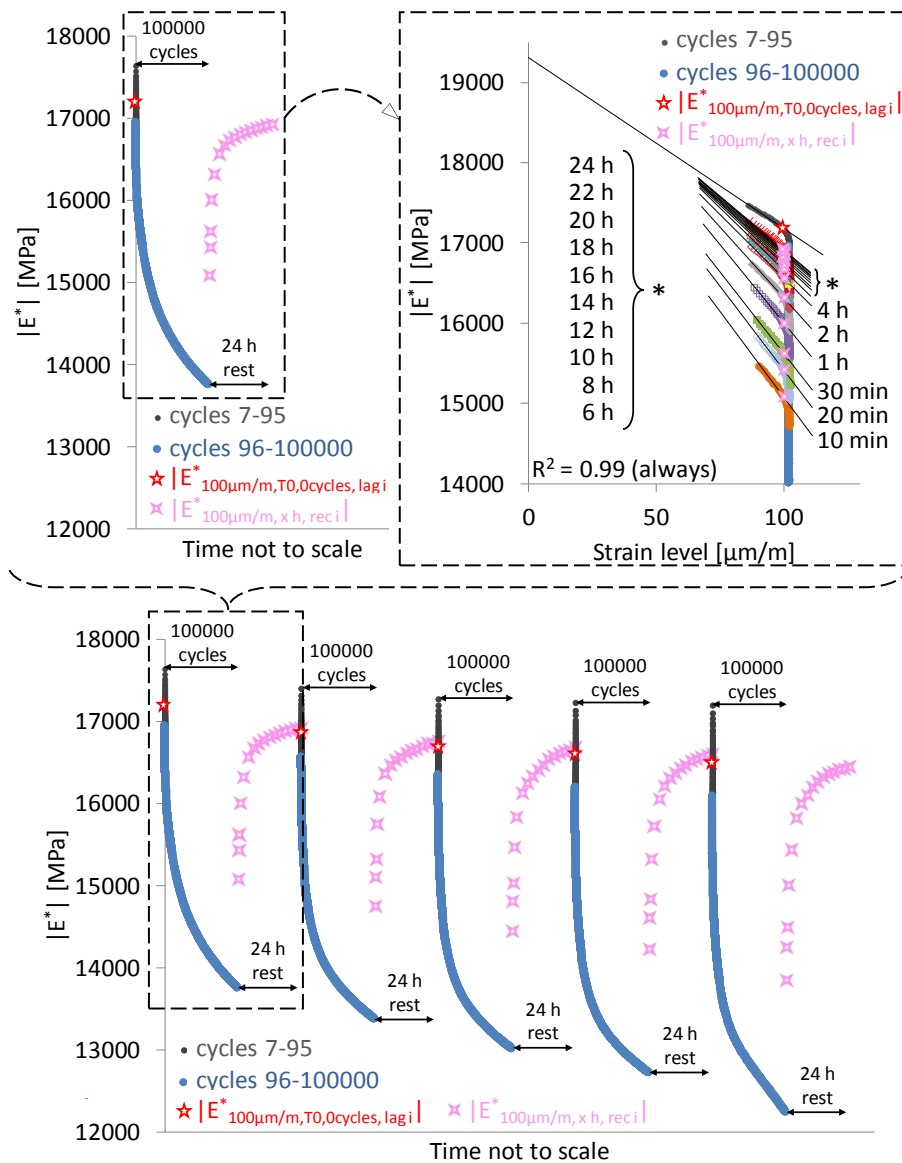


Figure 7: Results of Partial Fatigue Tests (PFTs) for mixture 35/50 B + 20% RAP: $|E^*|$ variation during fatigue lags and recovery periods. Stars indicate $|E^*_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}|$ calculated for each i th fatigue lag with its own non-linearity envelope. Complex modulus recovery is monitored using $|E^*_{100\mu\text{m}/\text{m}, x\text{ h}, \text{rec } i}|$ values, estimated at 100 $\mu\text{m}/\text{m}$, after x hours of recovery, with non-linearity envelopes.

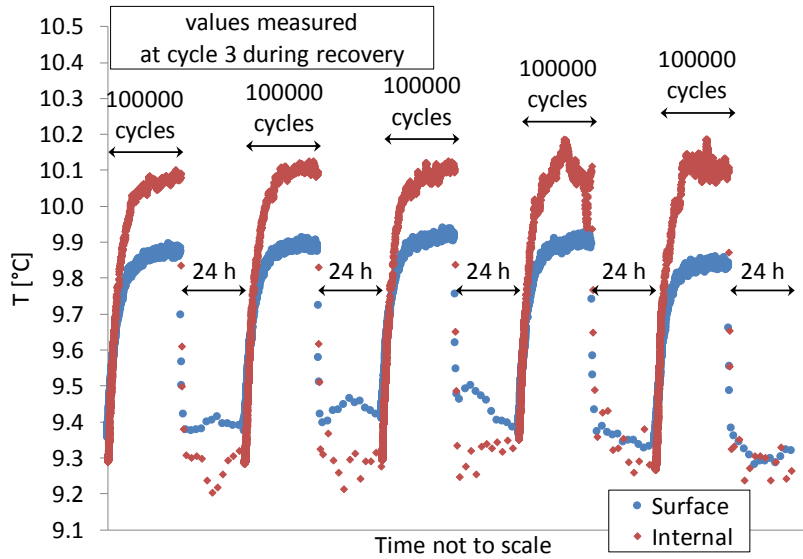


Figure 8: Results of Partial Fatigue Tests (PFTs) for mixture 35/50 B + 20% RAP: variation of internal and surface temperature during fatigue lags and recovery periods

Internal and surface temperature of samples were measured throughout all fatigue lags and recovery periods (Figure 8) shows values obtained for mixture 35/50 B + 20% RAP). Internal temperature was always taken into account for the analysis. A significant increase of internal temperature was observed during fatigue lags. Once a thermal equilibrium is reached between self-heating of the specimen (due to viscous energy dissipation) and heat exchange on the surface, a quasi-stationary temperature is observed. During rest periods, samples cool down relatively rapidly to the conditioning temperature. No significant self-heating is observed during short complex modulus tests performed during recovery periods, because of the small number of cycles applied (100 cycles). Therefore, temperatures measured at cycle 3 of each complex modulus test were considered.

Biasing effects and unrecovered variations of norm of complex modulus were quantitatively estimated from experimental results. For any *i*th lag, the influence of non-linearity, $\Delta|E^*_{\text{nonlinearity}}|$, was calculated as the difference between $|E^*_{0\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}|$ and $|E^*_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}|$ values:

$$\Delta|E^*_{\text{nonlinearity}}| = \left| |E^*_{0\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}| - |E^*_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}| \right| \quad (2)$$

The unrecovered variation of norm of complex modulus at the end of the 24 hour rest period for any *i*th fatigue lag, $\Delta|E^*_{\text{unrecovered } 24 \text{ h}}|$, was estimated with respect to the $|E^*_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}|$ value of the same lag, considered as the reference norm of complex modulus at 100 $\mu\text{m}/\text{m}$ in the undamaged condition, as in Equation (2):

$$\Delta|E^*_{\text{unrecovered } 24 \text{ h}}| = \left| |E^*_{100\mu\text{m}/\text{m}, 24\text{h}, \text{rec } i}| - |E^*_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}| \right| \quad (3)$$

where $|E^*_{100\mu\text{m}/\text{m}, 24 \text{ h}, \text{rec } i}|$ is the value of norm of complex modulus obtained at the end of the 24 hour rest period.

The influence of self-heating, $\Delta|E^*_{\text{heating}}|$, was considered as a function of the decrease of internal temperature of the sample during the rest period. In order to estimate the corresponding variation of norm of complex modulus, the b_E coefficient obtained with the CMTs (Equation (1)) was used, as follows:

$$\Delta|E^*_{\text{heating}}| = b_E \Delta T \quad (4)$$

where ΔT is the internal temperature difference, for any *i*th lag, between the value at the end of the fatigue lag (100,000th cycle) and the value during the last complex modulus test at the end of the 24 hour rest period.

Finally, the rest of the recovered $|E^*|$ variation not explained by self-heating is considered to be the effect of thixotropy. This hypothesis still needs validation. Further research work is ongoing on the subject. The influence of thixotropy, $\Delta|E^*_{\text{thixotropy}}|$, is estimated as in Equation (5):

$$\Delta|E^*_{\text{thixotropy}}| = \left| |E^*_{100000\text{cycles}, \text{lag } i}| - |E^*_{100\mu\text{m}/\text{m}, 24\text{h}, \text{rec } i}| - |E^*_{\text{heating}}| \right| \quad (5)$$

where $|E^*_{100000 \text{ cycles}, \text{lag } i}|$ is the last value of norm of complex modulus obtained at the end of any *i*th fatigue lag (100,000th cycle).

When comparing between them the non-linearity envelope lines obtained for all fatigue lags, for the same material, it can be observed that:

$$\left| E_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag \#1}}^* \right| > \left| E_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag \#2}}^* \right| > \dots > \left| E_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag \#5}}^* \right| \quad (6)$$

$$\left| E_{0\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag \#1}}^* \right| < \left| E_{0\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag \#2}}^* \right| < \dots < \left| E_{0\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag \#5}}^* \right| \quad (7)$$

It is important to note that, as already explained, both $|E_{0\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}^*|$ and $|E_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}^*|$ values are estimated according to non-linearity envelopes obtained with linear regression on experimental data having high R^2 values. However, while $|E_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}^*|$ are very close to experimental points, $|E_{0\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}^*|$ are extrapolated. For this reason, the comparison between non-linearity envelopes was performed based on $|E_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}^*|$ values. In particular, the difference between the non-linearity envelope of any i th lag and the envelope of lag #1, $|E^*|_{\text{envel. diff.}}$, was calculated as follows:

$$|E^*|_{\text{envel. diff.}} = \left| E_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}^* \right| - \left| E_{100\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag \#1}}^* \right| \quad (8)$$

Figure 9 shows an example of the estimation of biasing effects and unrecovered $|E^*|$ variations during fatigue lags #1 and #2 for mixtures 35/50 B + 20% RAP.

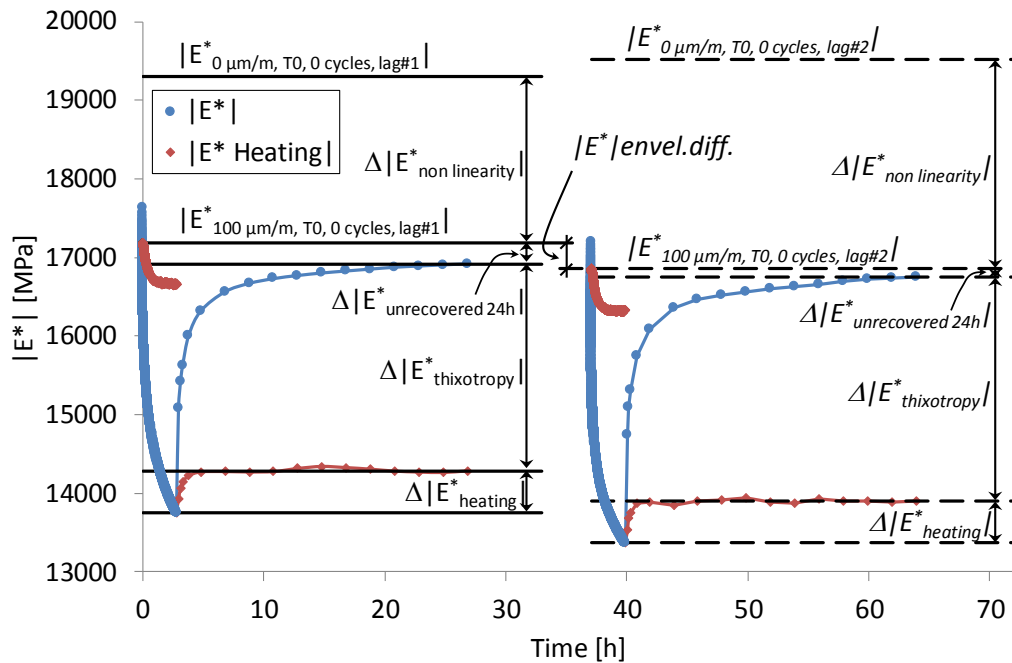


Figure 9: Estimation of biasing effects and unrecovered $|E^*|$ variations during fatigue lags #1 and #2 for mixtures 35/50 B + 20% RAP, using a different non-linearity envelope for each fatigue lag.

For each fatigue lag, the total $|E^*|$ variation $\Delta|E^*_{\text{total}}|$ was calculated as follows:

$$\Delta|E^*_{\text{total}}| = -\Delta|E^*_{\text{nonlinearity}}| + \Delta|E^*_{\text{unrecovered 24h}}| + |E^*_{\text{heating}}| + |E^*_{\text{thixotropy}}| = \left| E_{100000\text{cycles}, \text{lag } i}^* \right| - \left| E_{0\mu\text{m}/\text{m}, T_0, 0\text{cycles}, \text{lag } i}^* \right| \quad (9)$$

The relative contributions of all biasing effects and unrecovered variations of $|E^*|$ were then calculated with respect to total $|E^*|$ variations, for each fatigue lag. As an example, histograms of absolute and relative values obtained for mixture 35/50 B + 20% RAP are shown in Figure 10.

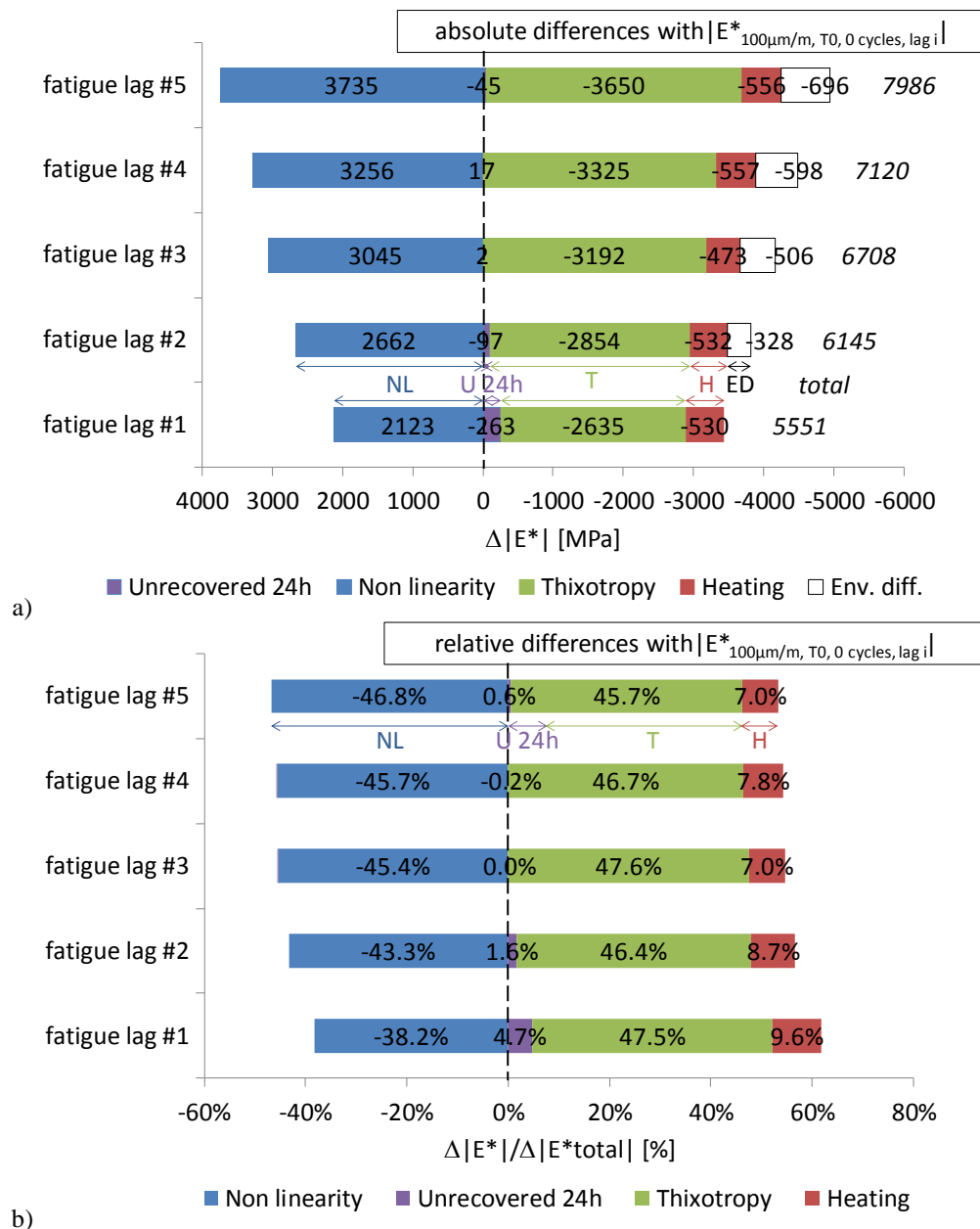


Figure 10: Estimation of biasing effects and unrecovered $|E^*|$ variations during fatigue lags #1 and #2 for mixtures 35/50 B + 20% RAP, using a different non-linearity envelope for each fatigue lag: (a, top) absolute values; (b, bottom) relative values

A second approach was also used to quantify biasing effects and unrecovered $|E^*|$ variations in a different way. Instead on calculating a different non-linearity envelope for each fatigue lag, the envelope of lag #1 was used as a reference to estimate all $|E^*|$ variations occurring during successive lags. Therefore, only the beginning of the first fatigue lag is retained as a true undamaged condition. Using this approach, by definition, the effect of non-linearity $\Delta|E^*_{\text{nonlinearity,ref:lag\#1}}|$ is constant throughout the five lags, since it is calculated only with the non-linearity envelope of lag #1, as in Equation (10):

$$\Delta|E^*_{\text{nonlinearity,ref:lag\#1}}| = |E^*_{0\mu\text{m}/\text{m}, T_0, 0 \text{ cycles, lag\#1}}| - |E^*_{100\mu\text{m}/\text{m}, T_0, 0 \text{ cycles, lag\#1}}| \quad (10)$$

Unrecovered variations $|E^*_{\text{unrecovered 24 h, ref:lag\#1}}|$ are calculated as

$$\Delta|E^*_{\text{unrecovered 24 h, ref:lag\#1}}| = |E^*_{100\mu\text{m}/\text{m}, 24 \text{ h, rec } i}| - |E^*_{100\mu\text{m}/\text{m}, T_0, 0 \text{ cycles, lag\#1}}| \quad (11)$$

Self-heating and thixotropy effects are identical to the ones calculated with the previous approach (Equations (4) and (5)), since they do not depend on the non-linearity envelope. Figure 11 shows an example of the estimation of biasing effects and unrecovered $|E^*|$ variations during fatigue lags #1 and #2 for mixtures 35/50 B + 20% RAP, using this approach.

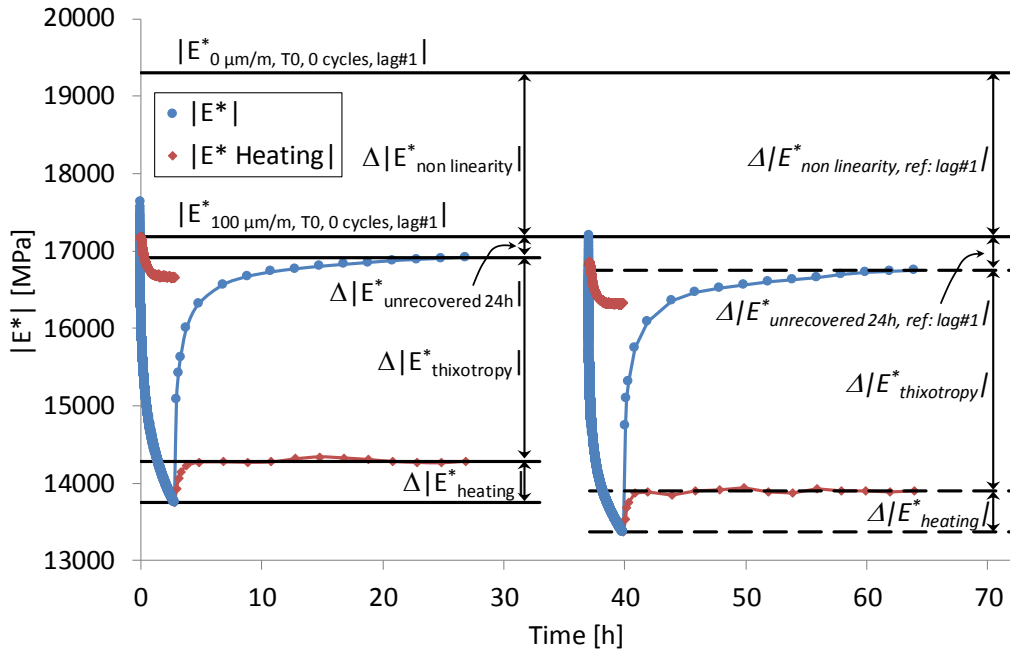


Figure 11: Estimation of biasing effects and unrecovered $|E^*|$ variations during fatigue lags #1 and #2 for mixtures 35/50 B + 20% RAP, using the non-linearity envelope of fatigue lag #1 as a reference for all lags.

Following the second approach, the total $|E^*|$ variation, $\Delta|E^*_{total,ref:lag\#1}|$, is calculated as:

$$\begin{aligned} \Delta|E^*_{total,ref:lag\#1}| &= -\Delta|E^*_{nonlinearity,ref:lag\#1}| + \Delta|E^*_{unrecovered\ 24h,ref:lag\#1}| + |E^*_{heating}| + |E^*_{thixotropy}| = \\ &= |E^*_{100000cycles,lag\ i}| - |E^*_{0\mu m/m,T_0,0cycles,lag\#1}| \end{aligned} \quad (12)$$

Similarly to what previously shown, the relative influence of all biasing effects and unrecovered variations of norm of complex modulus were calculated also with this approach, with respect to $\Delta|E^*_{total,ref:lag\#1}|$. Histograms of absolute and relative values estimated for mixture 35/50 B + 20% RAP by taking the non-linearity envelope of fatigue lag #1 as a reference are shown in Figure 12, as an example.

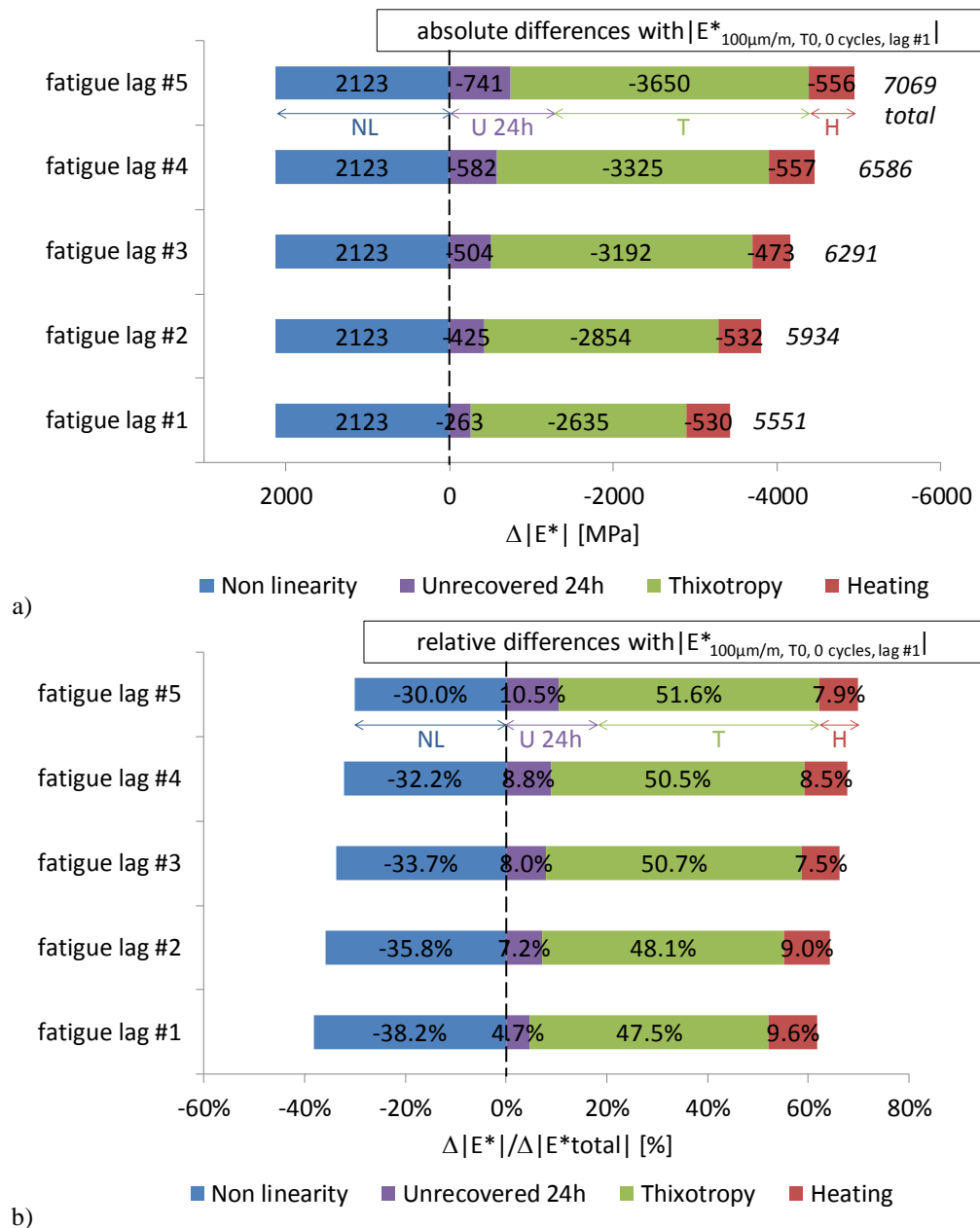


Figure 10: Estimation of biasing effects and unrecovered $|E^*|$ variations during fatigue lags #1 and #2 for mixtures 35/50 B + 20% RAP, using a different non-linearity envelope for each fatigue lag: (a, top) absolute values; (b, bottom) relative values

Because of the length limitation of the article, results obtained for all tested mixtures cannot be reported in detail. Mixture 35/50 and sample A of mixture 35/50 + 20% RAP failed during fatigue lag #3. Since failure occurred outside the region monitored by extensometers, the possibility that recorded data are not representative of real material properties cannot be excluded. Sample B of mixture 35/50 + 20% RAP also failed during fatigue lag #3, but failure occurred at the edge of the extensometer area. Test on mixture 35/50 + 2.5% SBS + 20% RAP was interrupted during fatigue lag #2 because of an excessive difference observed between strain amplitudes obtained by the three extensometers. These data indicate that the strain field within the sample was not homogeneous, therefore data obtained for this material during PFTs cannot be considered as representative of material properties. No problems were encountered during tests on mixtures 35/50 + 40% RAP, 35/50 B + 20% RAP and Orthoprène® + 20% RAP. Therefore, the following conclusions were drawn based on the results obtained for these three materials:

- The effects of self-heating on $|E^*|$ variations during fatigue tests are smaller than the effects of non-linearity and thixotropy. In particular, depending on the material, self-heating appears to account for about 7% to 16% of total $|E^*|$ variations during single fatigue lags. Approximately the same relative influences are found when overall

variations during the entire test are considered (that is, when $|E^*|$ variations are estimated with respect to the non-linearity envelope of fatigue lag #1).

- Using the new analysis procedure proposed in this paper, unrecovered variations of $|E^*|$ are substantially lower (half or less) than what found with the original procedure ([8]). This observation holds true both when each fatigue lag is examined separately and when the non-linearity envelope of lag #1 is used as a reference.
- The effects of non-linearity and thixotropy appear to depend on the tested material. In fact, the three considered mixtures show different relative proportions of these two biasing effects. In particular, the influence of non-linearity varies, approximately, from 20% for mixture 35/50 + 40% RAP to 55% for mixture Orthoprène® + 20% RAP. On the contrary, thixotropy has the highest influence on mixture 35/50 + 40% RAP, where it accounts for 60% or more of total $|E^*|$ variation, and the lowest influence on mixture Orthoprène® + 20% RAP, for which its impact is approximately equal to 30%.
- The most important conclusion is unrecovered variations of $|E^*|$ are extremely small for all materials. In the authors' opinion, unrecovered variations of the order of magnitude of 10^1 MPa cannot be taken into account, since they can be confounded with the experimental variability of the results. This explains also why during some fatigue lags a negative value of unrecovered variation is found (meaning that at the end of the recovery period the obtained $|E^*|$ is higher than at the beginning of the corresponding fatigue lag). Therefore, when considering each fatigue lag separately, only fatigue lag #1 and fatigue lag #2 show non-negligible unrecovered variations of $|E^*|$. When the non-linearity envelope of fatigue lag #1 is used as a reference, the overall unrecovered $|E^*|$ variation at the end of the whole procedure (500,000 cycles) is equal or lower than 10%. This finding confirms the need for a deep rethinking of current procedures for fatigue tests and analysis. Furthermore, the study of the self-healing phenomenon of bituminous materials cannot overlook a clear distinction between recovery of biasing effects after cyclic loading and restoration of material integrity after real damage occurrence.

5. CONCLUSIONS

A new treatment method is proposed for the results of the recently developed ALFABET (Advanced Laboratory Fatigue And Biasing Effects Test) experimental procedure. The main improvement concerns the evaluation of complex modulus during recovery periods, leading to a more reliable estimation of material properties without the influence of biasing effects (non-linearity, self-heating and thixotropy). When different bituminous mixtures were subjected to five 100,000 cycle fatigue periods (each followed by a 24 hour rest period), more than 95% of the $|E^*|$ variation observed during each period was completely recovered after the successive rest period. When considering the overall $|E^*|$ variation during the 500,000 cycles, less than 10% of it was unrecovered after the last rest period. These findings highlight the need for careful estimation of the self-healing phenomenon in bituminous materials. In particular, a clear distinction must be made between recovery of biasing effects and restoration of material properties after damage (cracking).

REFERENCES

- [1] Fatigue of bituminous mixtures, H. Di Benedetto, C. de la Roche, H. Baaj, A. Pronk and R. Lundström, *Materials and structures*, 37, 202-216, 2004. DOI: 10.1007/BF02481620
- [2] Prediction of mix modulus and fatigue law from binder rheology properties, N. Boussad, P. DesCroix, and A. Dony, *Journal of the Association of Asphalt Paving Technologists*, 65, 40-72, 1996.
- [3] Mixture and mode-of-loading effects on fatigue response of asphalt-aggregate mixtures, A. A. Tayebali, J. A. Deacon, J. H. Coplantz, T. John, and C. L. Monismith, *Journal of the Association of Asphalt Paving Technologists*, 63, 118-151, 1994.
- [4] Étude de la fatigue des enrobés bitumineux à l'aide du manège de fatigue du LCPC, C. de la Roche, J.-F. Corté, J.-C. Gramsammer, H. Odéon, L. Tiret, and G. Caroff, *Revue générale des routes et aérodromes*, 716, 62-74, 1994.
- [5] A question of fatigue?, M. E. Nunn, in *Performance and durability of bituminous materials*, J. G. Cabrera and J. R. Dixon (Eds.), 45-54, London: E & FN Spon, 1994.
- [6] Fatigue damage for bituminous mixtures: a pertinent approach, H. Di Benedetto, M. A. Ashayer Soltani, and P. Chaverot, *Journal of the Association of Asphalt Paving Technologists*, 65, 142-158, 1996.
- [7] Nonlinearity, heating, fatigue and thixotropy during cycling loading of asphalt mixtures, H. Di Benedetto, Q. T. Nguyen, and C. Sauzéat, *Road Materials and Pavement Design*, 5(Supp 1), 163-202, 2011. DOI: 10.1080/14680629.2004.9689992
- [8] Quantification of Biasing Effects during Fatigue Tests on Asphalt Mixes: Non-Linearity, Self-heating and Thixotropy, S. Mangiafico, C. Sauzéat, H. Di Benedetto, S. Pouget, F. Olard and L. Planque, *Road Materials and Pavement Design*, Advance online publication. DOI: 10.1080/14680629.2015.1077000
- [9] The temperature dependence of relaxation mechanisms in amorphous polymers and other glass-forming liquids, M. L. Williams, R. F. Landel and J. D. Ferry, *Journal of American chemical society*, 77, 3701-3707, 1955.