

# Stress and strain dependence of bituminous materials viscoelastic properties

Lucas Babadopulos<sup>1,2</sup>, Gabriel Orozco<sup>1</sup>, Salvatore Mangiafico<sup>1</sup>,  
Cédric Sauzéat<sup>1</sup>, Hervé Di Benedetto<sup>1</sup>

<sup>1</sup> *Université de Lyon, École Nationale des TPE (LGCB), LTDS (CNRS UMR 5513). Rue Maurice Audin, 69518 Vaulx-en-Velin CEDEX, France,*

[lucas.babadopulos@entpe.fr](mailto:lucas.babadopulos@entpe.fr), [gabriel.orozco@entpe.fr](mailto:gabriel.orozco@entpe.fr), [salvatore.mangiafico@entpe.fr](mailto:salvatore.mangiafico@entpe.fr),  
[cedric.sauzeat@entpe.fr](mailto:cedric.sauzeat@entpe.fr), [herve.dibenedetto@entpe.fr](mailto:herve.dibenedetto@entpe.fr)

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[lucsbaba@det.ufc.br](mailto:lucsbaba@det.ufc.br)

## ABSTRACT

Bituminous materials are considered as linear viscoelastic for a small number of applied cycles at low strain amplitudes. Their behaviour can be represented considering the complex modulus and the complex Poisson's ratio (isotropic case), which are obtained for sinusoidal loading. These two properties are independent of strain/stress amplitude in the case of linear viscoelasticity (LVE). However, when increasing amplitude, nonlinearity induces a strain dependence of the measured complex modulus. This paper investigates the phenomenon of nonlinearity in bitumen, mastic and bituminous mixture (BM). For bitumen and mastic, shear tests were performed on hollow cylinder specimens with the Annular Shear Rheometer (ASR) device, developed at University of Lyon/ENTPE. For BM, axial tension-compression tests on cylindrical specimens were carried out. Both tests are homogeneous and particularly adequate for nonlinearity investigation. Strain amplitude sweep (SAS) tests were performed at different temperatures and frequencies. Results show that BM presents non-negligible nonlinearity even for very small strain (few tens of  $\mu\text{m}/\text{m}$ ). Nonlinearity effect's intensity is temperature- and frequency- dependent. The LVE limit (strain at which 5% modulus decrease is obtained) decreases with temperature for BM and increases for bitumen and mastic. While mastic seems to inherit nonlinearity from bitumen, a secondary contribution from the granular skeleton may also play a role in BM nonlinearity.

**Keywords:** complex modulus, Linear ViscoElasticity (LVE), strain/stress amplitude, nonlinearity, LVE limit.

## 1. INTRODUCTION

Nonlinearity in bituminous materials is a reversible phenomenon that causes material stiffness to be dependent on the applied strain level (Di Benedetto et al., 2011 [1]). Doubbaneh (1995) [2] demonstrated the relevance of nonlinearity on the measurements of complex modulus (a stiffness property). In the referred work, nonlinearity was found to be relatively more important at higher temperatures and lower frequencies (stiffness could decrease of about 27% at 41°C and 1Hz when varying strain amplitude - half of peak-to-peak strain - from 30 to 80 $\mu\text{m}/\text{m}$ ). It is of practical use to define a parameter called Linear Viscoelastic (LVE) limit, corresponding to the strain level up to which the variation of the measured complex modulus is small (lower

1 than 5%, for example, [3]). Airey et al. (2003) [3] determined LVE limits for bitumen and  
2 bituminous mixtures, commonly used in the literature. While bitumen LVE limits varied  
3 considerably (from about 1 to 10% shear strain, depending on temperature and frequency),  
4 bituminous mixtures LVE limits appeared to be approximately constant (around 100 $\mu$ m/m axial  
5 strain). Other works demonstrated the importance of nonlinearity of bituminous materials [4-6].

6 It is to be observed that complex modulus is a linear viscoelastic property, defined for  
7 perfectly sinusoidal loading and response signals. This is the case when the input signal is  
8 sinusoidal and the material behaviour is linear. Since nonlinearity is being investigated,  
9 rigorously, the complex modulus could not be defined. However, for the case of small  
10 nonlinearities (no appreciable deviation of the obtained signals from sinusoidal functions) it is  
11 useful to define the “equivalent” complex modulus (Babadopulos, 2017, [7]). This parameter is  
12 obtained following the same procedure as for the classical complex modulus: after fitting  
13 sinusoidal functions to stress and strain signals, its norm is obtained by dividing the stress  
14 amplitude by the strain amplitude (cf. Section 2.4). The equivalent complex modulus at very  
15 small strain amplitudes, where the behaviour can be considered as LVE, is confounded with the  
16 “linear” complex modulus. Then, the equivalent complex modulus can be used to evaluate the  
17 effect of nonlinearity, by observing its change in comparison to the asymptotic “linear” complex  
18 modulus. This is done in this paper for bitumen, mastic and bituminous mixture. The paper aims  
19 at characterising the phenomenon of nonlinearity and determining LVE limits for the referred  
20 materials to investigate their relationship.

## 21 2. EXPERIMENTAL INVESTIGATION

22 Strain-controlled sinusoidal tests were used for mechanical characterisation. Classical  
23 complex modulus tests and Strain Amplitude Sweep (SAS) tests were performed to characterise  
24 the linear and nonlinear behaviour of the tested materials. Complex modulus tests use constant  
25 and sufficiently small strain amplitude, giving the “linear” complex modulus (near its asymptotic  
26 value at 0 $\mu$ m/m). Meanwhile, SAS tests involve applying different strain amplitudes, in order to  
27 determine how the equivalent complex modulus evolves with strain amplitude at fixed  
28 temperature and frequency.

29 For all tests, hydraulic presses were used. For bitumen and mastic, the Annular Shear  
30 Rheometer (ASR) test set-up (cf. Section 2.2) was used. For bituminous mixture, a tension-  
31 compression (T-C) test set-up was used. Details can be found in Babadopulos (2017) [7].

32 The ASR applies approximately homogeneous shear to the tested material, allowing easy  
33 interpretation of results when a nonlinear response is obtained. It is to be observed that this is not  
34 the case for non-homogeneous tests such as the plate-plate test geometry classically used for  
35 bitumen and mastic testing in the Dynamic Shear Rheometer (DSR). Since the tension-  
36 compression test set-up also produces theoretically homogeneous states of stress and strain, only  
37 homogeneous tests were performed in this work.

### 38 39 2.1 Materials and Tests

40 Pure straight-run bitumen (B5070) with 55dmm penetration (NF EN 1426, [8]) and 49.4°C  
41 softening point (NF EN 1427, [9]) was used in this work. The mastic (M5070\_30pc40-70) is a  
42 mix of this bitumen with spherical silica glass beads, with 30% in volume of particles, whose  
43 radius is comprised between 40 and 70 $\mu$ m (for 90% of the total mass of particles). The  
44 bituminous mixture (BM1) is a French EME (*Enrobé à Module Elevé*, High Modulus Asphalt)  
45 with limestone aggregate and 5.1% bitumen content (Phan et al., 2017, [10]).

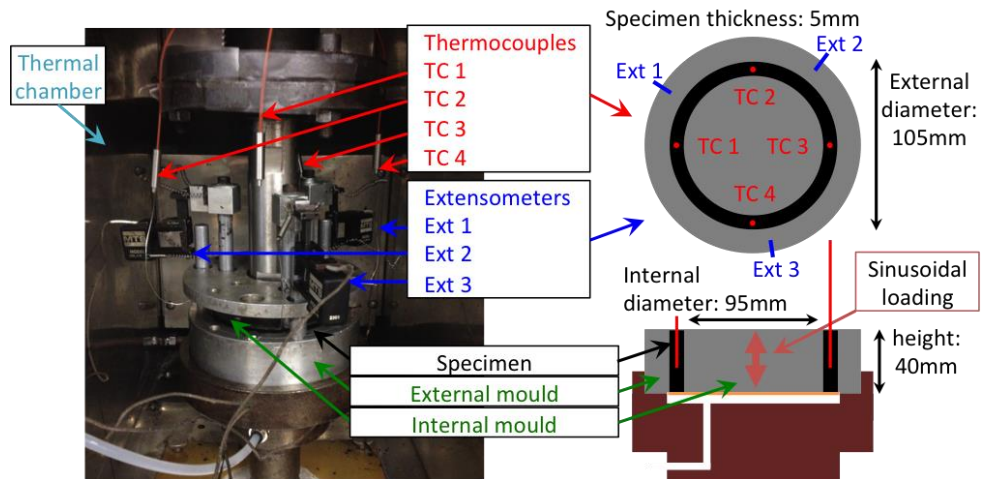
1 Table 1 presents an overview of the tested materials and the performed experiments. It also  
 2 includes the applied ranges of temperature and frequency. While for bitumen and mastic more  
 3 common SAS tests have been performed, for bituminous mixture a modified procedure was  
 4 utilised (Mangiafico et al., 2017, [11]). In this procedure, the strain amplitude is continuously  
 5 changed (during 50 loading cycles) between 0 and the targeted maximum strain amplitude. This  
 6 gives information on equivalent complex modulus for a more complete set of tested strain  
 7 amplitudes.

8  
 9 **TABLE 1 Overview of the experiments performed on bitumen (B5070), mastic**  
 10 **(M5070\_30pc40-70) and bituminous mixture (BM), with details on the tested temperatures**  
 11 **and frequencies**

Test set-up	Material/Specimen	Complex Modulus ( $E^*$ , or $G^*$ )	Strain Amplitude Sweep (SAS)
ASR	B5070_A	-3.2 to 25.4°C, 0.01 to 10Hz	-
ASR	B5070_B	-3.2 to 25.4°C, 0.01 to 10Hz	-
ASR	B5070_C	-10.0 to 25.4°C, 0.01 to 10Hz	-
ASR	B5070_F	-	6.3°C, 0.01 to 10Hz
ASR	B5070_G	-	11.1°C, 0.01 to 10Hz
ASR	M5070_30pc40-70_A	-9.8 to 25.5°C, 0.01 to 10Hz	-
ASR	M5070_30pc40-70_B	-9.8 to 25.5°C, 0.01 to 10Hz	-
ASR	M5070_30pc40-70_C	-	-3.2 to 15.0°C, 0.01 to 10Hz
T-C	BM1_C	-25 to 50°C, 0.003 to 10Hz	-10.0 to 14.0°C, 0.1 to 10Hz

12  
 13 **2.2 Annular shear tests on bitumen and mastic**

14 Four thermocouples (type T) immersed into the specimen were used to measure in-  
 15 specimen temperature, as presented in Figure 1. Three extensometers were used to measure shear  
 16 strain. Measured mean shear strain was used to control the loading to the desired strain path.  
 17



18 **FIGURE 1 ASR test set-up, and details on the test specimen with mounted sensors**

19  
 20  
 21 **2.3 Tension-compression tests on bituminous mixtures**

22 The tension-compression (T-C) test set-up used in this work is the same as Mangiafico et al.  
 23 (2017) [11]. A temperature probe (Pt100) adhered to the surface of the specimen was used to  
 24 measure the specimen temperature. Three extensometers were used to measure on-specimen  
 25 axial strain. Measured mean axial strain was used to control the loading to the desired strain path.

## 2.4 Experimental analysis

Fitting of sinusoidal functions (using least squares method) is used to determine stress amplitude ( $\tau_0$  for ASR tests and  $\sigma_0$  for T-C tests) and strain amplitude ( $\gamma_0$  for ASR tests and  $\varepsilon_0$  for T-C tests) for all cycles applied during the tests. Then, for each cycle, the norm of equivalent complex modulus can be determined as in Eq.(1) for ASR tests and as in Eq.(2) for T-C tests.

$$|G^*| = \frac{\tau_0}{\gamma_0} \quad (1)$$

$$|E^*| = \frac{\sigma_0}{\varepsilon_0} \quad (2)$$

In accordance with the obtained experimental results (cf. example in Section 3), it is supposed that an asymptotic norm of “linear” complex modulus exists. Its value is obtained by studying the evolution of equivalent complex modulus with the applied strain amplitude. Its definition is given in Eqs.(3,4), for ASR and T-C configurations, respectively.

$$|G_0^*| = \lim_{\gamma_0 \rightarrow 0} |G^*| \quad (3)$$

$$|E_0^*| = \lim_{\varepsilon_0 \rightarrow 0} |E^*| \quad (4)$$

With the previous parameters, it is possible to determine the LVE limits. This is done by assuming that the LVE limit is the strain level for which a 5% change in norm of equivalent complex modulus is obtained, as in Eqs.(5,6) for ASR and T-C configurations, respectively.

$$\text{LVE limit (shear strain amplitude)} = \gamma_{0,95\%} = \gamma_0(|G^*| = 0.95|G_0^*|) \quad (5)$$

$$\text{LVE limit (axial strain amplitude)} = \varepsilon_{0,95\%} = \varepsilon_0(|E^*| = 0.95|E_0^*|) \quad (6)$$

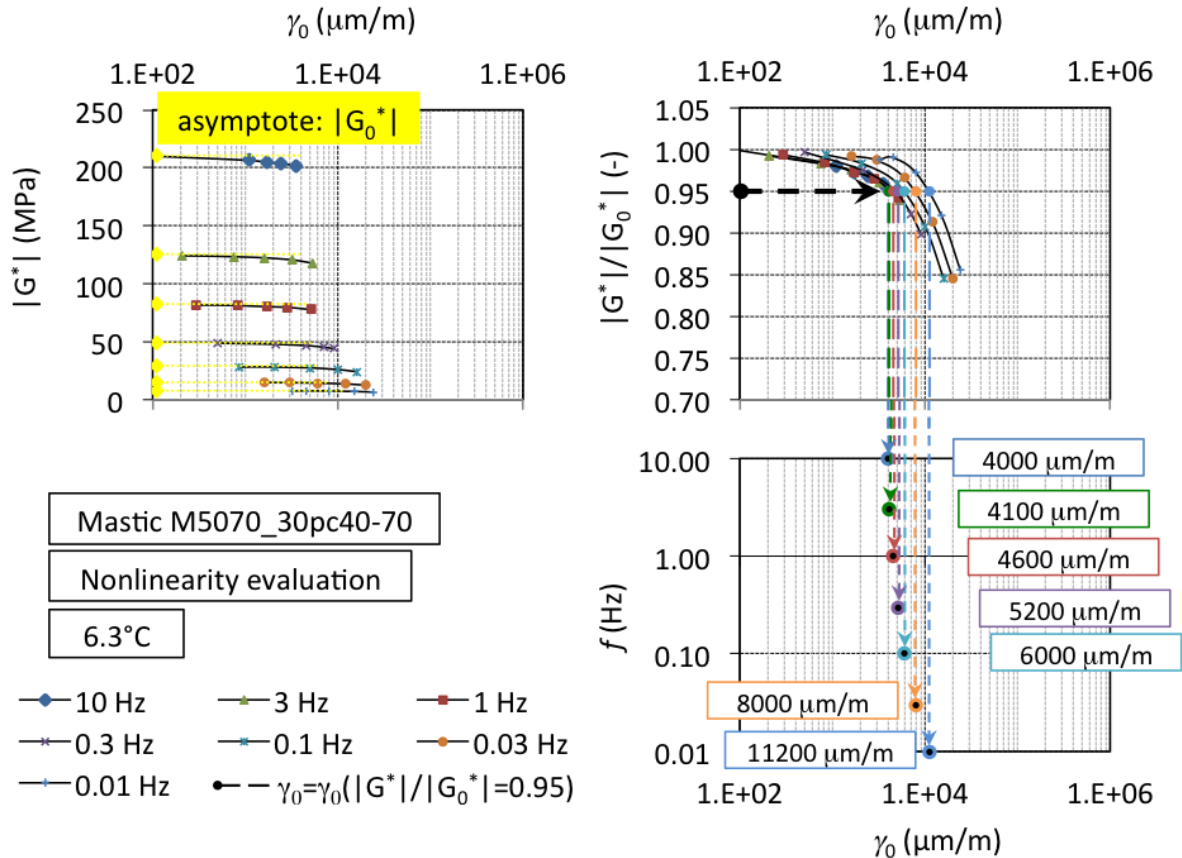
The time-temperature superposition principle, which may apply for the obtained experimental results, can be translated by the Williams-Landel-Ferry (WLF) equation. This equation is represented by Eq.(7).

$$\log a_T(T) = -\frac{c_1(T-T_{ref})}{c_2+(T-T_{ref})} \quad (7)$$

## 3. RESULTS AND ANALYSIS

### 3.1 Linear Viscoelastic (LVE) limits determination: example for mastic tested with the ASR

Figure 2 presents an example of analysis of the evolution of norm of equivalent complex modulus in order to obtain the LVE limits for various frequencies at a given temperature. The example is for the characterisation of mastic at 6.3°C with the ASR. The upper left corner presents the results norm of complex modulus and the determination of its asymptotic value at 0µm/m. In the upper right corner, norm of complex modulus results at each of the tested shear strain amplitudes were divided by the asymptotic “linear” value of norm of complex modulus. In this diagram, it is possible to determine the LVE limit by setting the relative change in equivalent complex modulus (in this paper, 5%), as shown in the figure. Then, in the down right corner, the results of LVE limits are represented for different shear strain amplitudes. The same procedure is applied for all the tested conditions evaluated in this work.



1  
 2 **FIGURE 2 Example of experimental results of norm of complex shear modulus from**  
 3 **ASR SAS tests (at 6.3°C on mastic M5070\_30pc40-70) and determination of asymptotic**  
 4 **“linear” values (at 0 $\mu\text{m/m}$ ) and of LVE limit in terms of strain amplitude ( $\gamma_{0.95\%}$ )**  
 5

### 6 3.2 Time and temperature dependence of LVE limits

7 From the classical complex modulus characterisation, WLF equation (Eq.7) constants were  
 8 obtained in order to represent the time-temperature superposition for the mechanical behaviour  
 9 of the tested materials. Table 2 presents the obtained values for these constants.

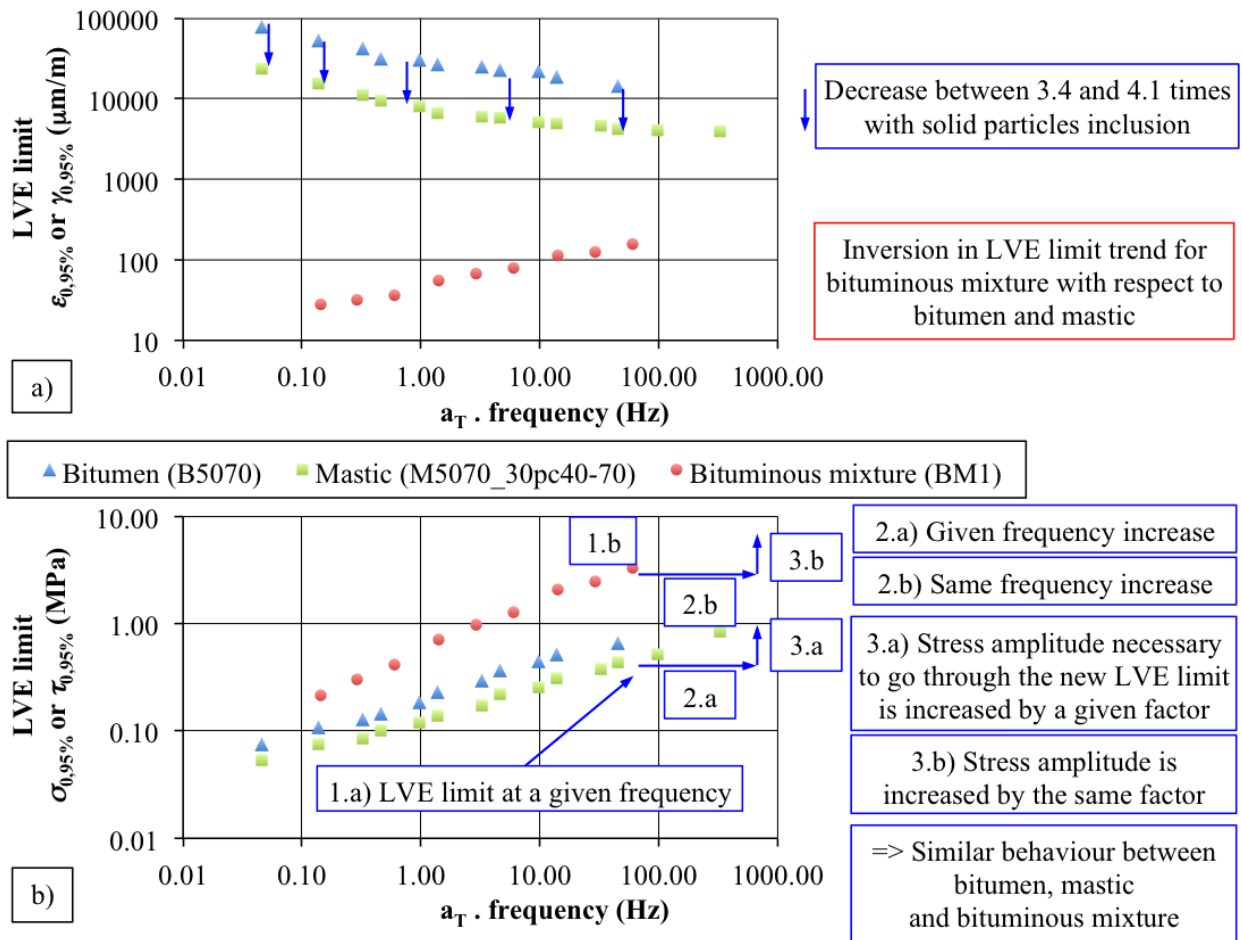
10 The LVE limits determined experimentally for each of the tested materials, at each of the  
 11 temperature and frequency conditions, are presented in Figure 3 as functions of the equivalent  
 12 frequency. The equivalent frequency was obtained after applying the time-temperature shift  
 13 factor (cf. Eq.7 and Table 2) using the same time-shift law as for the “linear” complex modulus  
 14 characterisation. While Figure 3a presents the LVE limits results in terms of strain amplitudes,  
 15 Figure 3b presents the results in terms of stress amplitudes. These are two ways of indicating the  
 16 effect of nonlinearity and they are intimately related by stiffness properties.

17 Regardless of the representation of LVE limits in terms of stress or strain amplitudes as  
 18 functions of equivalent frequency, it is seen that unique, smooth curves are obtained for each of  
 19 the materials. This indicates that the time-temperature superposition principle applies for the  
 20 LVE limit, and that the same time-shift behaviour as for the “linear” complex modulus is  
 21 obtained for this nonlinearity parameter. It is seen in Figure 3a that while the LVE limits in terms  
 22 of strain amplitudes decrease when increasing the frequency (or decreasing the temperature) for  
 23 bitumen and mastic, the inverse occurs for bituminous mixture. Also, the LVE limits for bitumen  
 24 and mastic seem to relate to each other by a simple factor for any given test condition. When

1 looking to the LVE limits in terms of stress amplitude, in Figure 3b, the inversion in trend is not  
 2 observed. Moreover, the lines obtained for all materials are approximately parallel. As explained  
 3 in the figure, when imposing a fixed change in the frequency, the LVE limit in terms of stress  
 4 amplitude changes following approximately the same factor for all materials. This indicates that  
 5 the time-temperature dependence of the nonlinearity effects on all materials is inherited from the  
 6 bitumen. For the bituminous mixture, the presence of a high volume fraction of particles  
 7 produces the differences in trend. The aggregate skeleton (which may also exhibit a non-linear  
 8 behaviour) is responsible for other differences.

10 **TABLE 2 WLF (Eq.7) constants used for time-shift of isotherms from classical complex**  
 11 **modulus characterisation for bitumen (B5070), mastic (M5070\_30pc40-70) and bituminous**  
 12 **mixture (BM1)**

WLF equation parameters			
Material	$T_{ref}$ (°C)	$C_1$ (-)	$C_2$ (°C)
B5070	15.0	31.2	188.6
M5070_30pc40-70	15.0	31.2	188.6
BM1	15.0	36.5	238.5



14 **FIGURE 3 Comparison of the determined LVE limits (in terms of axial or shear strain**  
 15 **amplitude,  $\epsilon_{0.95\%}$  or  $\gamma_{0.95\%}$ , figure a, and axial or shear stress amplitude,  $\sigma_{0.95\%}$  or  $\tau_{0.95\%}$ ,**  
 16 **figure b). The results are presented as master curves at 15°C**  
 17



## 1 4. CONCLUSION

2 This paper investigated the phenomenon of nonlinearity on bitumen, mastic and  
3 bituminous mixture. Classical complex modulus tests and strain amplitude sweeps tests were  
4 used. LVE limits were determined for all tested materials. It was shown that time-temperature  
5 dependence of LVE limits of bitumen, mastic and mixture follows the same shift factors as for  
6 their linear behaviour (as determined from complex modulus characterisation). Strain and stress  
7 LVE limits of mastic are proportional to the ones of bitumen for all tested equivalent frequencies.  
8 Strain LVE limits for these materials decrease with equivalent frequency, while stress LVE  
9 limits increase. For the bituminous mixture, both LVE limits increase with equivalent frequency.  
10 The presence of a high volume fraction of particles, and possibly a non-linear behaviour of  
11 aggregate skeleton, may be responsible for these differences.

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