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Fatigue properties of nano-reinforced bituminous mixtures: A viscoelastic continuum damage approach

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Abstract

The experimental investigation described in this paper focused on the effects of nanoclays on the fatigue behaviour of bituminous mixtures. Damage characteristics of a bituminous mixture produced by making use of a nano-reinforced binder were compared to those of a reference mixture obtained by employing the same neat bitumen used as a base in the preparation of the nanoclay–bitumen blend. Dynamic modulus tests and direct tension cyclic fatigue tests were carried out to determine the linear viscoelastic properties and the damage evolution characteristics of materials. Corresponding results were modelled by means of a viscoelastic continuum damage approach and by making use of a more empirical evaluation based on the classical Wöhler representation. It was found that the use of nanoclays produced a reinforcement of bituminous mixtures, the benefits of which were observed both in the progression of damage and in the occurrence of ultimate failure conditions.

Keywords: Bituminous mixture; Nanoclay; Fatigue; Viscoelastic continuum damage theory

1. Introduction

Fatigue damage in asphalt pavements is a complex phenomenon which occurs as a result of repeated traffic loading. Damage initiates with the formation of microcracks, then develops into larger visible cracks that appear on the pavement surface in the form of alligator cracking. Such a progression depends upon several factors related to mix composition, as well as to loading and environmental conditions [1].

Quantitative assessment of fatigue damage is a key issue that needs to be addressed both in the mixtures and pavement designs. Laboratory tests are required to simulate repeated loading in controlled conditions, and adequate models are necessary to interpret experimental data while taking into account the viscoelastic behaviour of these composite materials. In this context, rather than focusing exclusively on failure, the full damage process can be assessed by means of the viscoelastic continuum damage theory. Its effectiveness has been proven extensively as part of several research studies [2–7].

In order to enhance fatigue resistance of bituminous mixtures, use of modifiers and reinforcing agents is a valu-

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able design option. In this regard, a variety of innovative products and technologies have been proposed in recent years, in addition to traditional polymer-based compounds. In particular, reinforcement of mixtures based on the use of nano-sized additives represents a pioneering area of research which has already yielded encouraging results [8].

In general terms, when nano-sized additives are used for the preparation of composites, their very high surface-to-volume ratios generate a modifying action governed by the existence of surface contributions that prevail over bulk effects. Hence, in the specific case of bituminous mixtures, massive interactions which arise between nano-sized particles and surrounding bituminous binder can significantly improve the mechanical response of the final composite material [9].

Among the various possible nano-reinforcing agents, nanoclays are considered to be suitable for the development of bituminous nanocomposites [10]. Nanoclays are layered silicates in which sheets of nanometric thickness that constitute the mineral structure have the potential to separate from each other when incorporated into a generic matrix. When silicates are used in combination with organic matrices, a fundamental prerequisite to promote clay sheet separation is the modification of the character of clays from hydrophilic to hydrophobic. This is achieved by replacing the inorganic exchange cations which lie in the clay galleries with specific surfactant coatings that contribute to the expansion of galleries aside from changing the polarity of clays. Based on nanoscale interactions between silicate platelets and dispersing medium, nanocomposites can assume several degrees of intercalated and/or exfoliated configurations. Intercalated structures occur when the diffusion of matrix molecules within galleries leads to an expansion of interlayers between platelets, which still retain a predefined spacing. On the other hand, exfoliation results from diffusion phenomena that generate a complete separation and random distribution of clay sheets within the composite material [11,12].

The peculiar internal structures that can be generated by the morphological arrangement of nanoclays significantly impact the performance properties of composite materials. Several studies carried out on bituminous nanocomposites showed that clay layers provide an efficient anti-ageing action, which can be attributed to the combined effects of barrier properties of silicates and geometrical restraints due to the complex distribution of platelets [13–15].

Moreover, nanoclay modification has the potential to improve rutting performance due to the enhancement of stiffness and elasticity [16–19].

With regard to fatigue performance, differing experimental outcomes have been reported in literature. In fact, the resistance to withstand repeated loadings has been found to be strongly dependent on the physicochemical properties of base components, additive dosage and protocols used for the preparation of nano-reinforced binders [20–23]. Moreover, the choice of laboratory test conditions plays a major role in the fatigue performance exhibited by nanoclay-modified bituminous mixtures [24,25]. In spite of efforts to address this issue, the effect of nanoclays on fatigue properties of bituminous composites still needs further investigation.

The research work described in this paper investigates the effects of nanoclays on the damage characteristics of bituminous mixtures. Laboratory tests were carried out using the uniaxial configuration, and corresponding results were modelled by means of a viscoelastic continuum damage approach.

2. Materials and testing

Bituminous mixtures considered in the experimental study were produced by making use of a base binder (B) and of a reinforced binder (NB), originated by adding nanoclay to base binder B.

Base binder B was a PG 64-22 neat bitumen provided by Paramount Petroleum, Phoenix, Arizona. It was subjected to preliminary characterization which included determination of penetration at 25 °C [26], softening point [27], and viscosity at 135 °C [28]. Results of these tests are summarized in Table 1.

The nanoclay used for preparation of reinforced binder NB was a commercially available product supplied by BYK Additives & Instruments, Gonzales, Texas. It consists in a bentonite clay organically modified with bis(hydrogenated tallow alkyl)dimethyl salts used as surfactant coatings. Physical characteristics of the nanoclay, as reported in the manufacturer's specifications, are listed in Table 2.

Binder NB was prepared by following a protocol composed of the following subsequent steps. Base bitumen B was initially preheated for 1 h until it reached a target temperature of 130 °C, while the nanoclay was oven-dried for 1 h at 100 °C. Nanoclay was then incorporated into bitumen by manual mixing while keeping the tin containing the blend on a heating plate for 10 min. Based on results of previous studies, which focused on storage stability of nano-reinforced blends [29], the dosage of nanoclay was set equal to 3% by weight of base bitumen. The bitumen–nanoclay blend was then subjected to mechanical shear mixing using an Arrow 1750 Electric Mixer at a temperature of 125 °C. Mixing speed was maintained at 600 rpm for a total duration of 1.5 h. In order to avoid differences in thermal history that may have an effect on consequent physical and rheological properties, base binder B was subjected to the same processing protocol adopted for reinforced binder NB.

Table 1
Preliminary characterization of base bitumen B.

Penetration at 25 °C (dmm)	Softening point (°C)	Viscosity at 135 °C (Pa·s)
54	46.5	0.43

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Table 2
Physical characteristics of employed nanoclay.

Dry particle size (μm)	Packed bulk density (g/l)	Density (g/cm^3)	Basal spacing (nm)
<10	165	1.66	3.63

Bituminous mixtures were prepared in the laboratory by adopting a reference job mix formula characterized by the aggregate size distribution shown in Table 3, and by a binder content equal to 6.2% by weight of dry aggregates. Identification codes M-B and M-NB were associated with mixtures containing neat bitumen B and nano-reinforced binder NB, respectively. Mixture M-B was considered as a reference to which mixture M-NB could be compared to during the experimental investigation in order to highlight the effects caused by the use of nanoclay.

After aggregate-bitumen mixing and before mix compaction, loose blends were aged by spreading them in oven pans kept at a constant temperature of 135 °C for 4 h [30]. Cylindrical specimens with 150 mm diameter were then compacted to the target height of 180 mm by means of a gyratory compactor. Compaction temperatures were fixed at 147 °C and 155 °C for mixtures M-B and M-NB, respectively. Mass of the material inserted in the compaction mould was selected to obtain a final geometric air void content equal to $6.5 \pm 0.5\%$. Cylindrical specimens for mechanical testing were cored from gyratory specimens and their upper and lower portions were cut off by sawing.

Evaluation of damage characteristics of the bituminous mixtures was carried out by means of dynamic modulus tests and direct tension cyclic fatigue tests. Equipment used for both tests was a universal testing machine with maximum load of 25 kN.

Dynamic modulus tests were performed on specimens of 100 mm in diameter and 150 mm in height. Measurements were run according to AASHTO PP 61 [31] at temperatures of 4.4, 21.1 and 38 °C, and loading frequencies of 25, 10, 5, 1, 0.5 and 0.1 Hz. Average results obtained from three samples per mixture were considered in the analysis.

Direct tension cyclic fatigue tests were carried out according to AASHTO TP 107 [32] on specimens of 75 mm in diameter and 150 mm in height at a temperature of 18 °C. Such a temperature was determined as the average of the high and low PG extremes minus 3 °C of neat binder B. Tests were run in the strain controlled mode at three different strain levels (300, 350 and 400 μstrain). Five nominally identical specimens were tested for each mixture.

Table 3
Aggregate size distribution of bituminous mixtures M-NB and M-B.

Sieve	Size (mm)	Passing (%)
1″	25	100
3/4″	19	100
1/2″	12.5	92.8
3/8″	9.5	82.5
No. 4	4.75	56.8
No. 8	2.36	40.3
No. 16	1.18	30.0
No. 30	0.6	20.7
No. 50	0.3	12.5
No. 100	0.15	7.3
No. 200	0.075	2.0

3. Results and analysis

Preliminary screening of results obtained from uniaxial fatigue tests was done by considering failure modes. Results of two tests carried out on mixture M-B were discarded due to the occurrence of either edge failure or partial glue detachment. In comparison, all tests performed on specimens of mixture M-NB were considered valid because of the occurrence of failure in the middle portion of test cylinders, possibly as a result of lower mixture variability and better consistency in performing tests.

Raw data obtained from fatigue tests were analysed by means of the viscoelastic continuum damage (VECD) model which as an output yields pseudosecant modulus C as a function of damage parameter S (damage characteristic curve). The former parameter is a measure of material integrity ($C = 1$ for an undamaged material), while the latter is an internal state variable. *S-C* plots are of premium interest in the analysis of fatigue behaviour since they are material-specific and do not depend upon mode of testing (i.e., stress- or strain-controlled), temperature, loading frequency and stress or strain level [5].

Theoretical background of the VECD model is constituted by the elastic–viscoelastic correspondence principle, the work potential theory and the time–temperature superposition principle [33,34].

The elastic–viscoelastic correspondence principle is used to isolate viscoelastic effects from those related to damage [35]. By replacing physical strains with pseudostrains, the viscoelastic problem is reduced to a correspondent elastic problem in which time-dependant effects are eliminated. Pseudosecant modulus C can thus be calculated using Eq. (1):

$$C = \frac{\sigma}{\varepsilon^R \cdot I} \tag{1}$$

where σ is applied stress, ε^R is the corresponding pseudostrain and I is a specimen variability compensation parameter.

Shapery's work potential theory [36], based on thermodynamic principles which apply to irreversible processes, allows modelling of damage growth in viscoelastic materials according to the following damage evolution law (Eq. (2)):

$$\frac{dS}{dt} = \left(-\frac{\partial W^R}{\partial S}\right)^{\alpha} \tag{2}$$

where S is the damage internal state variable, α is the material-dependent damage evolution rate and W^R is the pseudostrain energy density function. This is defined in Eq. (3):

$$W^R = \frac{1}{2} \cdot \sigma \cdot \varepsilon^R = \frac{1}{2} \cdot \left(\varepsilon^R\right)^2 \cdot C(S) \quad (3)$$

Derivation of the internal state variable as a function of time can be obtained by combining Eq. (3) with Eq. (2) and by solving the resulting equation (Eq. (4)):

$$\frac{dS}{dt} = \left(-\frac{1}{2} \cdot \left(\varepsilon^R\right)^2 \cdot \frac{dC}{dS}\right)^{\alpha} \quad (4)$$

Different methods have been proposed and successfully used to solve the damage evolution law of bituminous mixtures in uniaxial loading conditions [37,38].

The time–temperature superposition principle is used to incorporate temperature effects in the VECD model by replacing actual time with reduced time at the given reference temperature [37,39]. Reduced time is calculated from actual time using the time–temperature shift factors determined as part of linear viscoelastic characterization.

If a rigorous VECD approach is used in the calculation of pseudostrain, pseudostiffness and damage by means of results obtained from direct tension fatigue tests and dynamic modulus tests, the entire loading history of cyclic fatigue data must be tracked, thus leading to a cumbersome calculation procedure. To overcome such a problem, as indicated in AASHTO TP 107 [32] a simplified approach can be followed, in which data retrieved from fatigue tests are separated into two parts. The first part, which com-

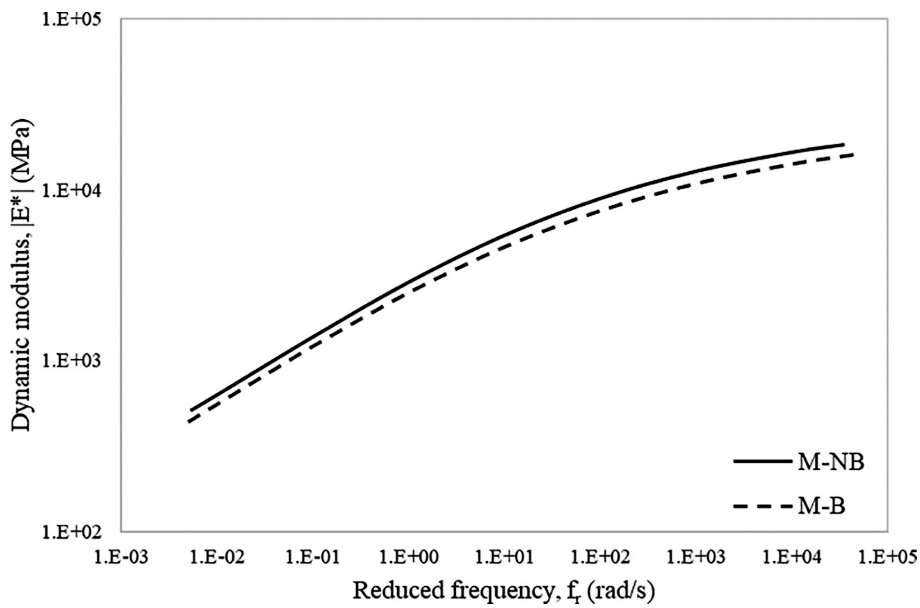

Fig. 1. Master curves of the dynamic modulus of bituminous mixtures M-NB and M-B.

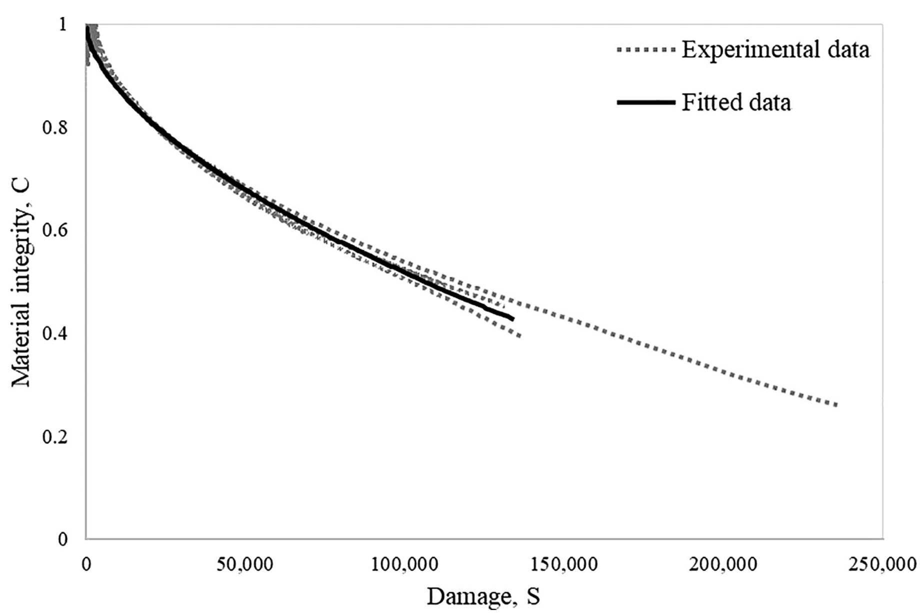

Fig. 2. Damage characteristic curves of bituminous mixture M-NB.

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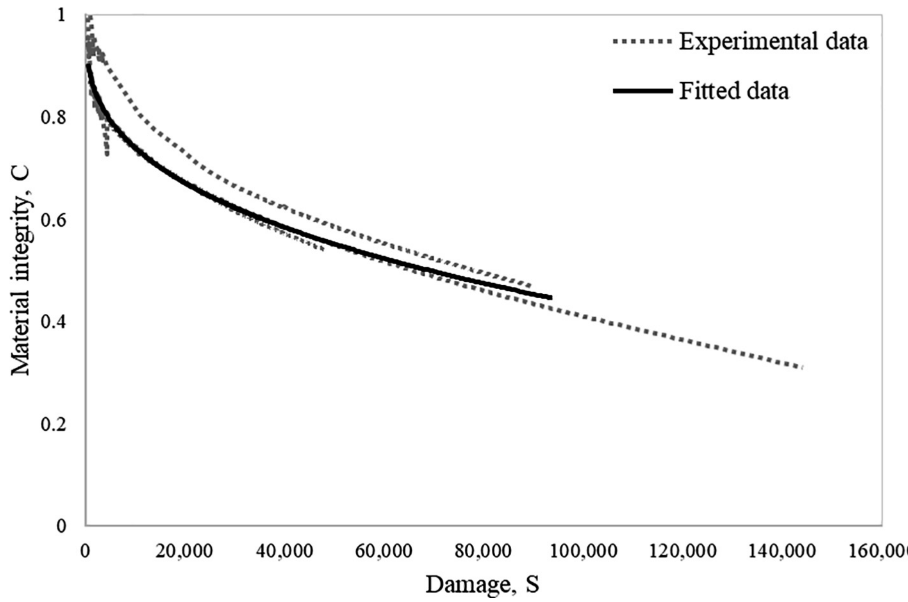

Fig. 3. Damage characteristic curves of bituminous mixture M-M-B.

prises data of the initial portion of the first loading cycle (from zero to first peak stress), is analysed using the rigorous time-step based damage formulation that involves determination of Prony coefficients of the relaxation modulus from dynamic modulus and phase angle values measured according to AASHTO PP 61 [31]. This is done since evolution of damage which occurs at the onset of loading plays a key role in the overall fatigue response and thus requires a detailed representation. The second part, which comprises the rest of data recorded during the fatigue test, is analysed in a simplified manner by referring to peak-to-peak pseudostrain calculated as the product of physical peak-to-peak strain and linear viscoelastic dynamic modulus at the specific testing temperature and frequency. In the present study, data processing was made by means of the ALPHA-F software (distributed

Table 4
C_{11} and C_{12} fitting parameters of bituminous mixtures M-NB and M-B.

Mixture	C_{11}	C_{12}	R^2
M-NB	5.770E−04	5.845E−01	0.970
M-B	1.160E−02	3.377E−01	0.969

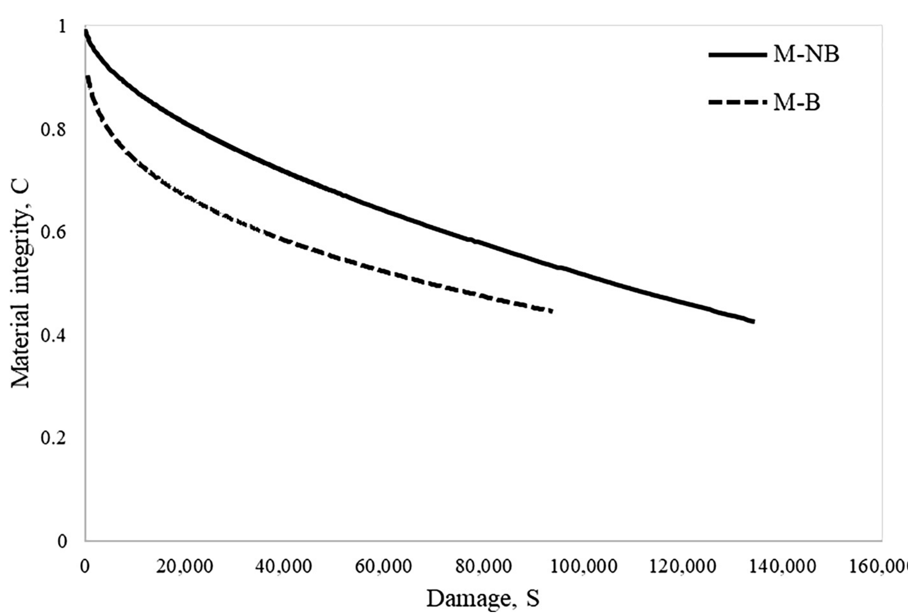

Fig. 4. Damage characteristic curves of bituminous mixtures M-NB and M-B.

by Instrotek Inc.) which adheres to the principles and procedures outlined above.

Results obtained from dynamic modulus tests were checked for replicate variability by referring to the maximum value of the coefficient of variation, which was equal to 0.062 and 0.059 for mixtures M-NB and M-B, respectively. Master curves were thereafter constructed by fitting average data to the shift factors function and sigmoidal function presented in Eqs. (5) and (6), respectively:

$$Loga_T = \alpha_1\left(T^2 - T_{ref}^2\right) + \alpha_2\left(T - T_{ref}\right) \quad (5)$$

$$Log|E^*| = \delta + \frac{\alpha}{1 + 1/e^{\beta+\gamma(Logfr)}} \quad (6)$$

Master curves at 20 °C are shown in Fig. 1, where they are plotted by considering only the range of reduced frequencies in which experimental data were available. It can be observed that for mixtures characterized by the same composition and geometric void content, nanoclay binder reinforcement produces a slight stiffening effect. Such a result is coherent with previous findings, which showed that the use of nanoclays leads, both for binders and mixtures, to an improvement of resistance to permanent deformation [16–19].

S-C damage data derived from the combined analysis of dynamic modulus and fatigue tests were fitted to the power law function reported in Eq. (7) [3]:

$$C = 1 - C_{11} \cdot S^{C_{12}} \quad (7)$$

where C_{11} and C_{12} are mixture-dependent fitting coefficients.

Data obtained from the VECD model and corresponding fitting curves are shown in Figs. 2 and 3. Table 4 lists the values of corresponding C_{11} and C_{12} constants and of coefficient of determination R^2.

A comparison between power law functions of the damage characteristic curves of the two mixtures is provided in Fig. 4. Functions have been plotted up to the average value of integrity parameter *C* recorded at failure (i.e., at peak phase angle) during fatigue tests.

It can be observed that damage progression is influenced by constitution of the binder phase. In the case of nano-reinforcement, material integrity is lost more gradually as a function of damage, especially in the early phases of repeated loading. Such an occurrence may be attributed to the advantageous redistribution of local stresses, which may take place in the presence of nanoclay arrangements within the bituminous binder.

Other observations can be made by considering the extension of damage curves in the investigated strain range. In the case of nano-reinforcement, failure conditions are associated to a level of damage ($S = 134{,}389$) which is higher than that of the mixture containing neat bitumen ($S = 93{,}786$). Corresponding conditions reached in terms of material integrity are also significantly different, with values of the *C* parameter equal to 0.43 and 0.45 for mixture M-NB and M-B, respectively. These outcomes seem to suggest that nanoclay particles while interacting with bitumen may locally act as barriers to microcrack propagation, thus causing fatigue ductility effects associated to an increase in endurable damage before failure.

The comparative analysis described above is coherent with the results obtained by making use of the classical Wöhler representation, in which strain amplitude imposed during fatigue testing (ε) is plotted as a function of the number of loading cycles to failure (N_f). This is shown in

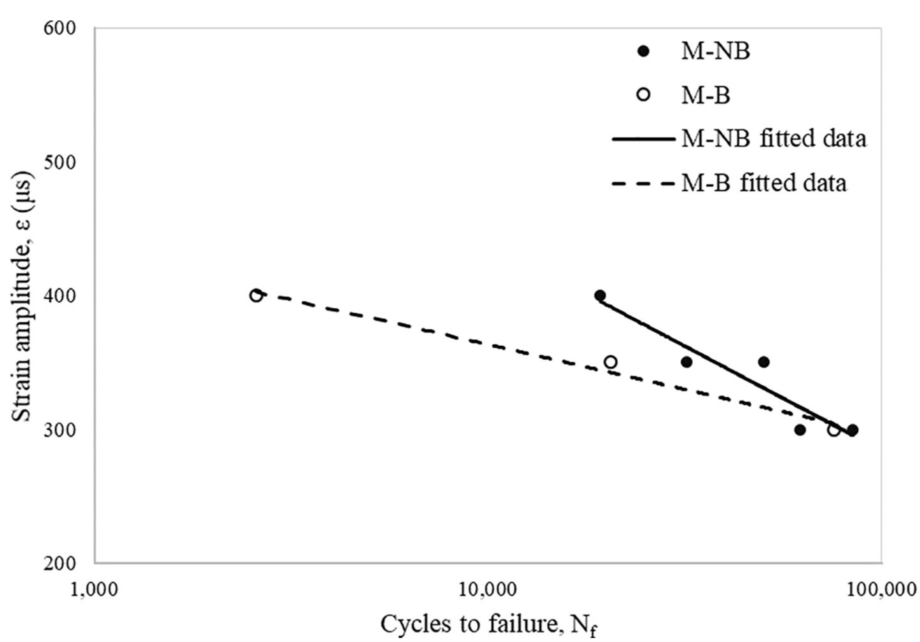

Fig. 5. Wöhler's fatigue curves of bituminous mixtures M-NB and M-B.

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Fig. 5, in which limiting fatigue lines were fitted to experimental data.

In Fig. 5 it can be observed that mixture M-NB containing the nano-reinforced binder exhibited a higher fatigue resistance than reference mixture M-B. Such a difference is especially relevant at higher strain values, while the behaviour of the two mixtures is approximately equivalent at lower strain levels. Obtained fitting lines suggest that an inversion of the relative ranking may occur at very low strain levels and high loading cycles, although such an extrapolation is not totally justified by the limited width of the investigated strain range.

4. Conclusions

Results obtained in the study described in this paper suggest that the use of binders containing nanoclays may improve the fatigue behaviour of bituminous mixtures. This was verified by considering the entire process of damage evolution by means of the viscoelastic continuum damage model. Further evidence was also found by means of a more empirical evaluation based on the classical Wöhler representation.

It was postulated that observed improvements may be due the redistribution of local stresses and to microcrack barrier effects caused by nanoclay arrangements, which form within the bituminous binder. These observations and explanations are compatible with typical nanoclay–bitumen interactions and are coherent with previous findings.

Future validation of the encouraging results outlined above should be supported by analyses carried out on a wider set of base materials, blends and mixtures. Furthermore, improvements may be sought by optimizing the protocol for preparation of nano-reinforced binders in order to better control the effective degree of intercalation and/or exfoliation of nanoclay platelets.

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