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Test Method

Developments in dynamic testing of rubber compounds: assessment of non-linear effects

G. Ramorino [a], D. Vetturi [a], D. Cambiaghi [a], A. Pegoretti [c], T. Ricco [b,*]

[a] *University of Brescia, Department of Mechanical Engineering, Via Branze 38, I25123 Brescia, Italy*
[b] *University of Brescia, Department of Chemistry and Physics for Engineering and Materials, Via Valotti 9, I25123 Brescia, Italy*
[c] *University of Trento, Department of Materials Engineering, Via Mesiano 77, I38050 Trento, Italy*

Abstract

In the present work, a test method to characterize the dynamic behaviour of rubber compounds by electrodynamic shaker (ES) in the frequency range of 10–1000 Hz was developed. Data of dynamic moduli of two different rubber compounds were determined through the analysis of the transmissibility of a suitably designed test system. The results were compared with those of dynamic moduli master curves obtained through frequency–temperature reduction of data measured by a commercial dynamic mechanical thermal analyser (DMTA), by scanning temperature at various frequencies in the range 0.3–30 Hz. Very good agreement of the data obtained by the two different aproaches were found, in spite of the different range of frequency explored by the two instruments, ES and DMTA, respectively. For one of the material examined, non-linear effects at low strain amplitudes were investigated by the two experimental methods considered.
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1. Introduction

In many applications the overall performance of rubber components is governed by the dynamic behaviour of the material of which they are constituted. For example, the dynamic properties of the material, such as stiffness and damping, determine the transmission of vibrations through rubber isolators or the rolling resistance and heat buildup in tyre treads.

As a consequence of the viscoelastic nature of rubbers, their dynamic and thermal behaviour is significantly dependent on frequency and temperature. The material stiffness and hysteretic effects, associated with the energy dissipation and consequent heating within the material, are usually described in terms of the complex dynamic modulus $E^* = E' + iE''$, E' and E'' being the storage and loss components, respectively, dependent on temperature and frequency of the applied load [1]. Further, due to the high content of carbon black usually incorporated together with other additives within the compounds to optimize the mechanical performances of the rubber components [2], the dynamic behaviour of these materials is markedly non-linear [3–12]. This implies that E' and E'' depend also on the deformation amplitude the material undergoes. Thus, in order to quantify the dynamic behaviour of a carbon filled rubber it is necessary to produce data with varying frequency, temperature and strain amplitude within ranges of interest.

Dynamic testing has made substantial progress in the two last decades by the use of electrodynamic forced vibration machines with high speed data acquisition systems and fully computer controlled. Today, extensive

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* Corresponding author: Tel.: +1-39-030-3715785; fax: +1-39-030-3715788.
  E-mail address: ricco@ing.unibs.it (T. Ricco).

characterization of the dynamic moduli of elastomeric materials is usually performed using commercial Dynamic Mechanical Thermal Analyzers (DMTA) that enable testing by scanning temperature and frequency. The tests are typically performed in tensile mode on small specimens up to a frequency of 50–100 Hz.

Tests on rubber components, even of considerable dimensions, can be performed by electrodynamic shakers (ES). These instruments enable the evaluation of the transmissibility of a rubber mounting with varying frequency in the range of about 10–1000 Hz.

In the present work a new test to measure the dynamic moduli of rubbers at high frequencies by electrodynamic shaker was developed. The tests were performed with varying frequency between 10 Hz and 500 Hz at different strain amplitudes (lower than 1%). The data were found consistent with the results of dynamic moduli master curves determined through frequency–temperature reduction of experimental data obtained by a DMTA apparatus, by scanning temperature at various frequencies in the range 0.3–30 Hz.

2. Experimental

2.1. Materials

The tests were performed on two different elastomeric compounds: sample A and sample B, kindly supplied by Pirelli SpA (Milan, Italy) and by CF Gomme SpA (Brescia, Italy), respectively. Sample A consisted of a vulcanized filled rubber containing carbon black and other additives, supplied in the forms of cylinders of 17.5 mm diameter and 24.7 mm height, and 1.9 mm thick sheets. The elastomeric phase was based on SBR/polybutadiene rubber compound and the black content was about 50 phr. Sample B consisted of an epoxidized natural rubber (ENR) containing about 17 phr carbon black and other additives, supplied in the form of both cylinders with 29.4 mm diameter and 25.0 mm height, and 2.0 mm thick sheets. More detailed information about the composition of the elastomeric compounds was not available from the manufacturers.

2.2. Testing by electrodynamic shaker (ES)

Dynamic tests were carried out using the apparatus sketched in Fig. 1a, and consisting of: (1) an electrodynamic shaker by L.D.S. Ltd (UK) driven by a computer-based controller in sinusoidal testing mode, (2) a testing device (see Fig. 1b) consisting of some cylindrical rubber specimens joined upwards to a steel ring, that acted as a suspended mass, and downwards to another steel ring fixed to the vibrating table (the number of the cylindrical rubber specimens adopted in the tests was six for sample A and four for sample B, respectively), (3) small mass

piezoelectric accelerometers to measure the acceleration of the vibrating table and the suspended mass, respectively, (two accelerometers were placed on the top of the suspended mass, and another one on the vibrating table), (4) a digital acquisition system to monitor and treat the signals. The cylindrical rubber specimens were joined to the upper and lower steel rings by suitable adhesives. The value of the suspended mass was controlled by an additional mass fixed on the top of the testing system: typically a total mass of 2 kg and 4 kg was adopted for sample A and B, respectively.

The tests were carried out at various frequencies in the range between 40 and 500 Hz under displacement control of the vibrating table, driven by a sinusoidal signal, so as to maintain a constant strain amplitude in the rubber specimens with varying frequency. The levels of the strain amplitude examined were 0.2, 0.5, and 0.7% for sample A, and 0.7% for sample B. The different levels of strain were obtained by varying the value of the suspended mass.

Mechanical hysteresis was found to produce temperature increase within the rubber test-pieces. For each set of tests the temperature increase was monitored by a small sensitive thermocouple embedded within one of the rubber cylinders, and kept under control so as to guarantee a constant test temperature with variations of $\pm 1°C$. For each test, conducted at a given frequency and strain amplitude, the response of the accelerometers was acquired for several periods of oscillation and mean values of amplitude and phase were considered. The acquisitions were obtained by the use of a 16 bit AD converter. The sensitivity of the accelerometers employed was 100 mVs^2/m.

2.3. Testing by DMTA

Dynamic tests were also carried out by a Dynamic Mechanical Thermal Analyser (DMTA) by Polymer Laboratories Ltd. (UK) which can perform dynamic testing by scanning temperature and frequency simultaneously. The specimens consisted of 40x5x1.9 mm^3 strips, cut from the sheets. The tests were carried out in tensile mode at different levels of the strain amplitude, i.e. 0.2%, 0.5%, 0.7%, 1.4% for sample A and 0.7% for sample B. Tests were performed at five different frequencies, 0.3, 1, 3, 10, 30 Hz by scanning temperature in the range from –80°C to +100°C, at a heating rate of 0.4°C/min.

3. Results and discussion

For sample B, experimental data of the storage and loss modulus, E' and E″, respectively, were obtained by DMTA by scanning temperature for the different frequencies examined at a strain amplitude of 0.7%. The
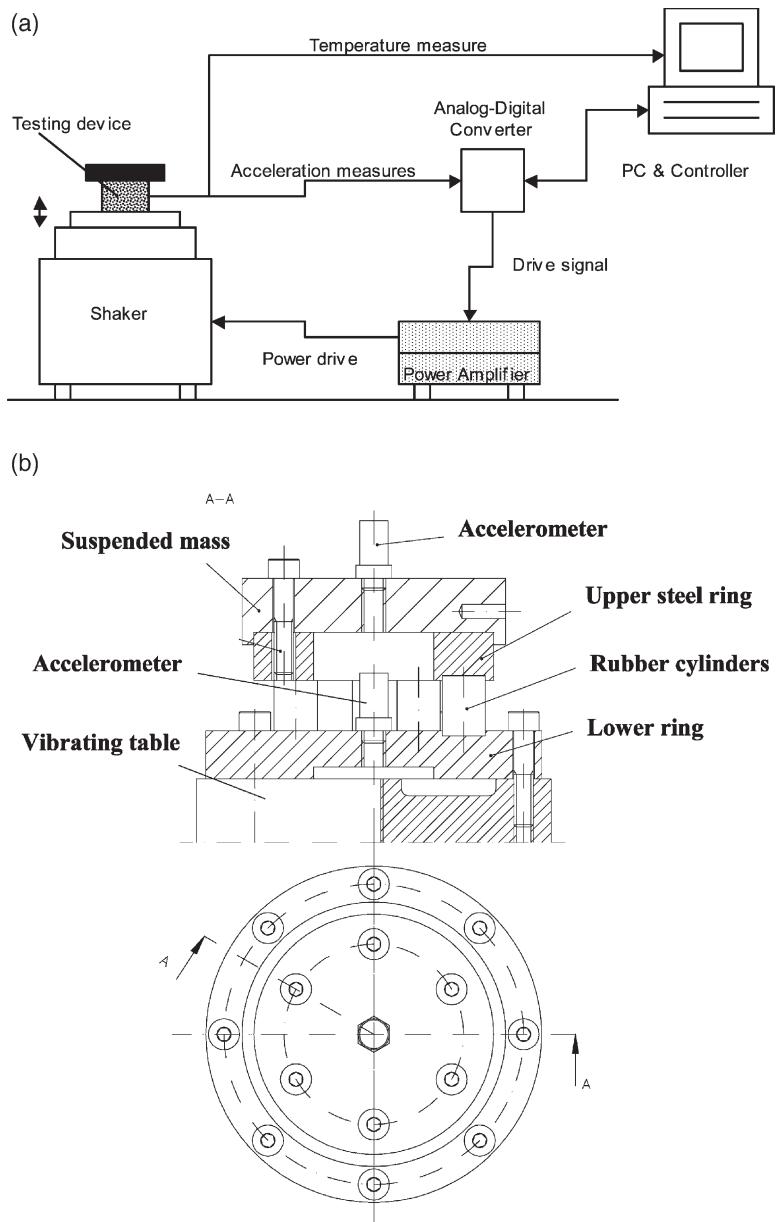
Fig. 1.   Scheme of the apparatus (a) and test system (b) used for dynamic testing by ES.

results are shown in Fig. 2a and b. The cross-plot of the curves of Fig. 2a allowed the construction of a series of isothermal curves for E' at various temperatures as a function of frequency in the range between 0.3 and 30 Hz, as shown in Fig. 3. These data were then reduced according to a frequency–temperature superposition approach (1) to obtain the master curve of the dynamic storage modulus E'(ω) as a function of frequency, ω, at the reference temperature of 20°C. Each experimental value of E' in the rubber-like region was previously reduced by the rubber elasticity factor T/T_0. Then the

isothermal curves were shifted horizontally to best super-position. The procedure provided an empirical determination of the shift factor a(T), that determines the position of the master curve on the frequency axis at any other given reference temperature. A series of isothermal master curves of E' referred to different temperatures in the range between 20 and 50°C are reported in Fig. 4a. The same approach, applied to the data of the loss modulus E'', provided the master curves of E''(ω) reported in Fig. 4b. It is worthwile noting that by this approach any possible thermorheological complexity of the material

(a)

(b)
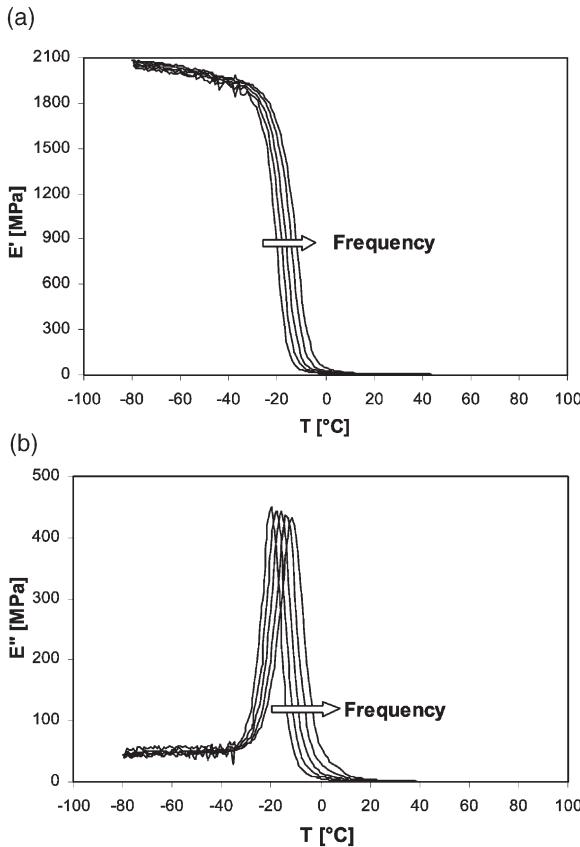
Fig. 2. Storage (a) and loss (b) modulus of sample B as a function of temperature measured by DMTA for a strain amplitude of 0.7% at various frequencies (0.3, 1, 3, 10, 30 Hz).
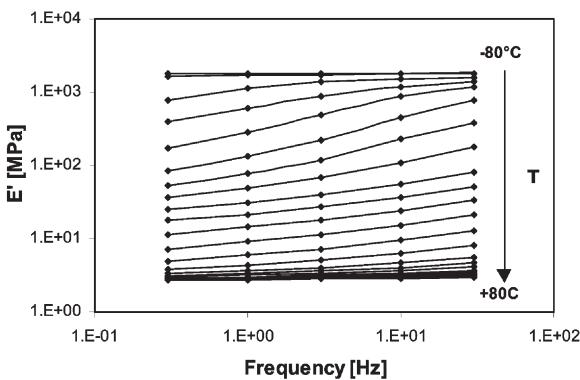
Fig. 3. Isothermal storage modulus values for sample B as a function of frequency, for a strain amplitude of 0.7%.

has been neglected. Indeed, thermorheological complexity has been observed for filled rubbers [13,14], mainly associated with the transition zone [14].

The treatment of the data obtained by ES was based on the assumpion that the test system can be modelled

(a)

(b)
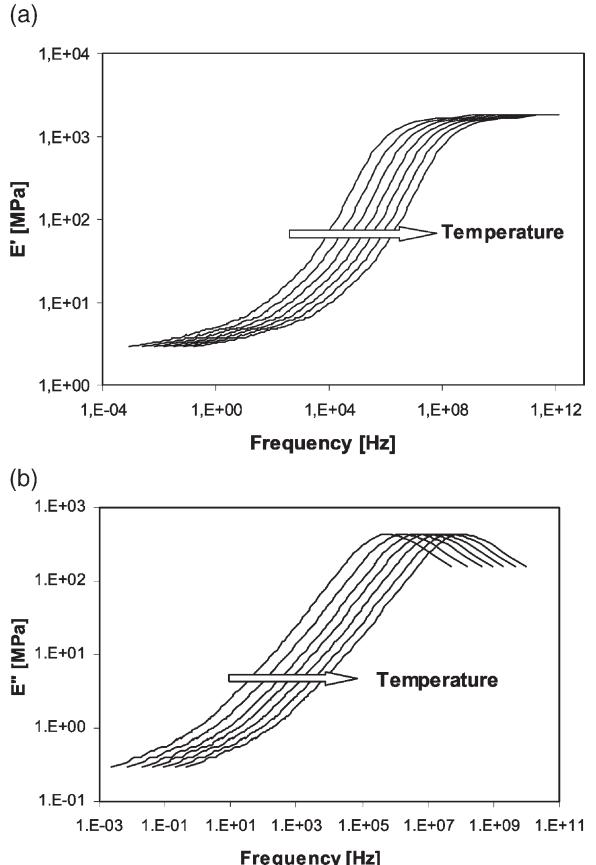
Fig. 4. Storage (a) and loss (b) modulus master curves of sample B for a strain amplitude of 0.7% at various reference temperatures (20, 25, 30, 35, 40, 45, 50°C).
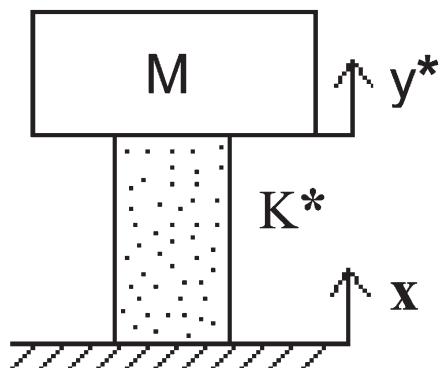
Fig. 5. Sigle degree of freedom system model.

by a single degree of freedom system with hysteretic damping, as shown in Fig. 5. This model is characterized by a mass, M, a complex spring rate $K^*(\omega) = K'(\omega) + iK''(\omega)$, and its dynamic behaviour is governed by the following equation:

$$M\ddot{y} + K^*(y-x) = 0 \qquad (1)$$

$\ddot{y}$ being the acceleration of the suspended mass M, and y and x the displacements of the suspended mass and the vibrating table, given by $x = x_0 e^{iwt}$ and $y = y_0^* e^{i\omega t}$, respectively. The complex trasmissibility of test system, $H^*(\omega)$, that represents the amplification/attenuation of vibrations from the vibrating table (input) to the suspended mass (response), can be expressed in the complex form as follows:

$$H^*(\omega) = H'(\omega) + iH''(\omega) = y/x = \ddot{y}/\ddot{x} \qquad (2)$$

$$= 1/2 \cdot (V_a + V_c)/V_b$$

H' and H'' being the real and imaginary part of transmissibility, respectively, $\ddot{x}$ the acceleration of the vibrating table, V_b and V_c the signals of the two accelerometers, both placed on the top of the suspended mass, and V_a that of the accelerometer placed on the vibrating table.

Substituting $\ddot{y} = -\omega^2 y$ in Eq. (1) and taking into account Eq. (2), the following relationship between H^* and K^* is obtained:

$$K^* = M\omega^2 H^*(\omega)/(H^*(\omega)-1) \qquad (3)$$

whereas the material dynamic modulus E^* and the complex spring rate are assumed to be simply related through a factor that accounts for the rubber specimen geometry:

$$K^* = sE^* \qquad (4)$$

For a resilient element of uniform cross-sectional area A_o and length l_o the following expression for s has been reported [15]: $s=(A_o/l_0)(1+\beta S^2)$, where S is a shape factor and β a numerical factor. It has been shown [15] that for a rubber cylinder of diameter D and height l_0, $S = D/4l_0$, and β can be taken to equal 2. For the vibrating system examined the number of the rubber cylinders was also considered.

For sample B the transmissibilty of the test system was measured by using Eq. (2) with varying frequency at a temperature of 23°C and at a strain amplitude of 0.7%. The results of the modulus of the complex transmissibility $Tr = |H^*| = (H'^2 + H''^2)^{1/2}$, simply named transmissibility, and the phase angle $\phi = arctg2(H',H')$ are reported as a function of frequency in Fig. 6. A peak of transmissibility is clearly observed at the resonance frequency of about 80 Hz. Changing the suspended mass shifts the curve, without any change in the shape, along the frequency axis as predicted by Eq. (3), since H^* is constant if $M\omega^2$ is constant. Successively, Eq. (3) and Eq. (4) were used to predict the values of T and ϕ from the data of the master curves of E' and E'' (Figs. 4a and 4b), obtained by DMTA. These values, also reported in Fig. 6, appear to be in very good agrement with those directly measured, showing the consistency of the approach adopted. Inversely, the data obtained by the electrodynamic shaker were converted to data of material
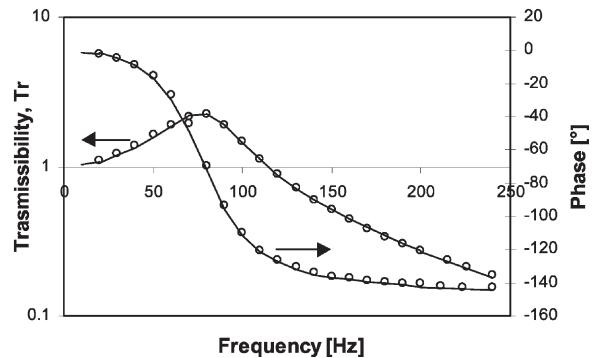
Fig. 6. Transmissibility and phase of the test system, adopted for ES testing, as a function of frequency. Rubber test-pieces of sample B. Data obtained by ES (full lines) and evaluated from dynamic moduli master curves obtained by DMTA (open circles).

dynamic modulus E^*. Such data for E' at temperatures of 23, 24, 25, 42, 50°C are reported in Fig. 7a together with the isotherm master curves previously obtained on the basis of DMTA experiments. Again, very good agreement between the data obtained with the two different methods can be observed. The same result has been obtained for E' data. This agreement also represents a validation of the model assumed for the vibrating system connected with the electrodynamic shaker and of the applicability of the frequency–temperature superposition principle, adopted to treat DMTA data.

Carbon black fillers are added to rubber to increase stiffness, abrasion resistance, tear strength and fatigue life. Their addition also increases both the rubber dynamic storage modulus and the phase angle, tgδ (tgδ = E''/E'), and these dynamic properties become dependent on strain amplitude [3–12]. The magnitude of these
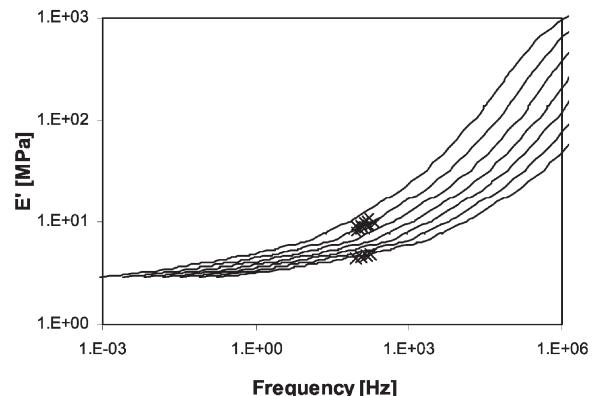
Fig. 7. Portion of storage modulus master curves of sample B at various reference temperatures (as shown in Fig. 4a). The values of E', determined from trasmissibility data obtained by ES testing at T = 25°C and T = 50°C are also reported (× symbol).

effects is determined both by the quantity and type of carbon black introduced into the compound. Non linear characteristics of the material response to dynamic input are generally found from very low levels of strain amplitude [8,11]. This behaviour of filled rubber is generally to referred to as the 'Payne effect' and a number of mechanism-based theories have been proposed to explain it [16–20]. Another possible source of non- linear effects is concerned with non-uniform strain field throughout the specimen, as in the case of specimen bulging in the compression mode. For this reason, non linear effects due to material should be studied in shear mode. However, due to the low levels of strain amplitude explored in the present work, we think that non linear effects due to specimen geometry can be reasonably neglected.

Non-linearity effects were observed and studied on sample A that contains a high amount of carbon black. (Due the low content of carbon black, sample B did not exhibit any substantial non-linearity). DMTA experiments were performed on sample A at different levels of strain amplitude, and at each strain level master curves of E' and tgδ (tgδ = E''/E') were obtained, following the same procedure previously described for sample B. Again, any possible thermorheological complexity of the material was neglected. The isometric master curves of E' and tgδ at the reference temperature of 22°C over a frequency range of several decades are reported in Figs. 8a and 8b, respectively. The shift factors, a(T), obtained from the construction of the master curves at the different strain amplitudes were found to be very similar. The non-linear effects are clearly shown by the decrease of the rubbery plateau of E' (low frequency region), and the increase of tgδ in the same frequency range, by increasing strain amplitude. Non-linear effects are not appreciable in the glass-like region at high frequencies. As also reported in a previous work [21], the tg δ master curves show that the viscoelastic transition peaks shift to lower frequencies, pointing out the viscoelastic feature of the non-linear effects. Recently, a deep insight about the viscoelastic character of Payne effect has been reported in the literature [22].

For the same material, dynamic experiments at three different levels of strain amplitude were also performed by ES with varying frequency at a temperature of 22°C. The effects of strain amplitude are observed on the hysteresis loops, such as those reported in Fig. 9, evaluated at a frequency of 140 Hz. A slight distortion from the elliptical shape can also be observed. The results for transmissibility Tr are shown in Fig. 10 as a function of frequency for three different strain amplitudes. It emerges that the resonant frequency and the value of transmissibility at the peak decrease with increasing strain amplitude. These findings, observed also by other authors [12], can be attributed to the decrease of the storage modulus E' with strain amplitude, which produces the decrease of the resonance frequency, and to the
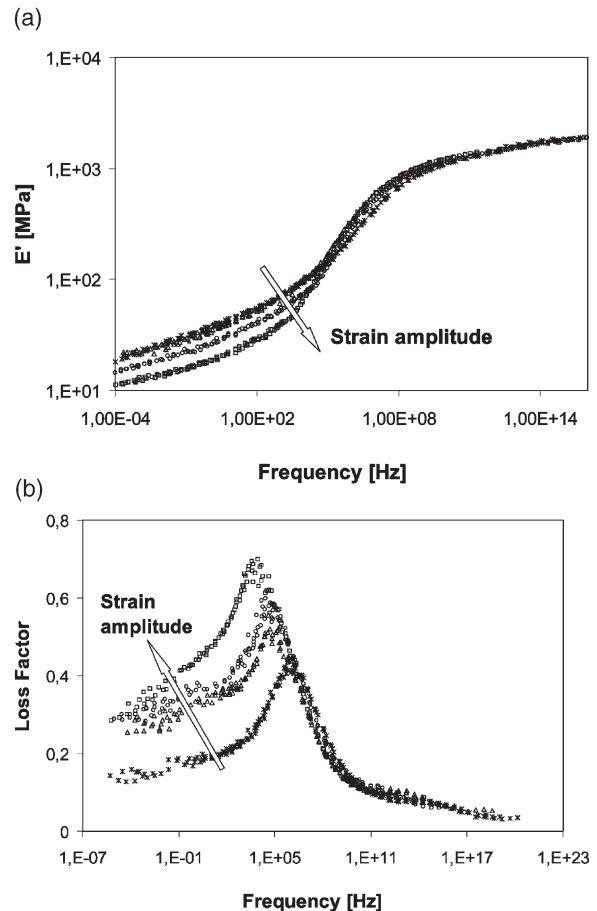
Fig. 8. Storage modulus (a) and loss factor (b) master curves of sample A for various strain amplitudes (×, 0.2; △, 0.5; ○, 0.7; □, 1.4%) referred to a reference temperature of 22°C.
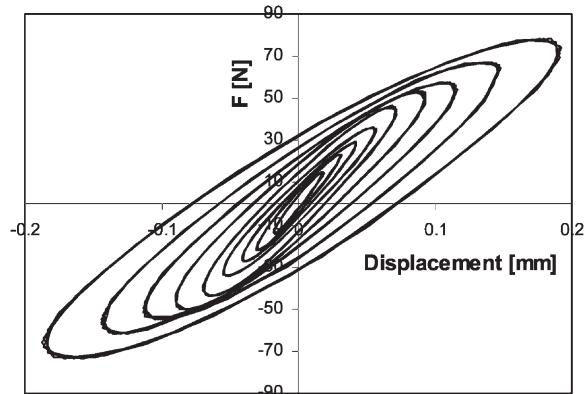
Fig. 9. Hysteresis loops for sample A determined by ES at various amplitudes of displacement.
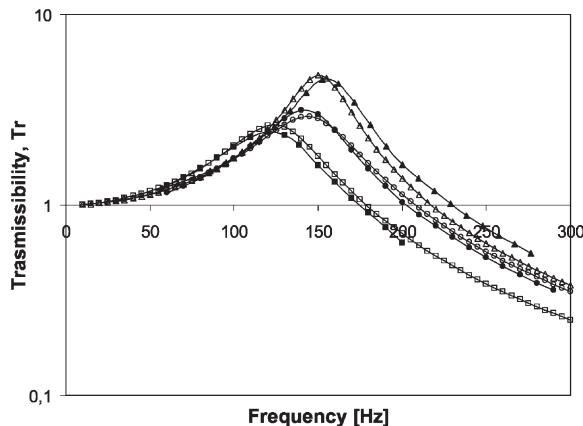
Fig. 10.   Transmissibility of the test system, containing rubber test pieces of sample A, as a function of frequency. Data determined by ES testing (full symbols) and by dynamic moduli obtained by DMTA (open symbols) at different strain amplitudes (▲△, 0.2; ●○, 0.5; ■□, 0.7%).

increase of the loss factor, tgδ, which causes higher damping.

The values of transmissibility of the test system used in ES experiments were also calculated from the data of E' and tgδ, obtained by DMTA and shown in Fig. 8a and b, using Eqs. (3) and (4). Strictly, Eq. (3) holds only for linear systems and its use for non-linear systems could lead to incorrect results; however, it has been hypothesised that the form of Eq. (3) could be maintained letting the nonlinearity be accounted for by the dependence of E*, and then of K*, on strain amplitude. The and b, using Eqs. (3) and (4). Strictly, Eq. (3) holds only approach are also reported in Fig. 10. Very good agreement with the direct measurements is found.

4. Concluding remarks

In the present work, a test method to characterize the dynamic behaviour of rubber compounds by an electrodynamic shaker (ES) in the frequency range of 10–1000 Hz was developed. Data of dynamic moduli of two different rubber compounds were determined through the analysis of the transmissibility of the test system, modelled by considering a single degree of freedom.

Non-linear effects (Payne effect) were also characterized for one of the two rubber compounds examined.

In parallel, the values of the storage modulus and loss factor were also experimentally determined by dynamic mechanical thermal analysis at various frequencies (in the range 0.3–30 Hz), temperatures and strain amplitudes by a commercial DMTA apparatus. For each strain amplitude level explored, master curves of the storage modulus and loss factor were obtained by frequency–temperature reduction scheme.

The results obtained clearly show that the test method, adopted to characterize the dynamic properties of filled rubbers by ES, produces reliable data consistent with those obtained by experiments carried out by DMTA, in spite of the different range of frequency attainable by the two instruments. Hence, this agreement represents a validation of the experimental procedure and the model assumed for the vibrating test system connected with ES, as well as of the applicability of the frequency–temperature reduction approach to obtain dynamic moduli master curves from DMTA data.

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