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EXPERIMENTAL MEASUREMENT OF THE DYNAMIC MODULI OF NATURALLY AGED RUBBER SEGMENTS

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Abstract: Subjects of experimental investigation were damping segments of Polyisoprene-Polybutadiene Rubber Blend (IR/BR) used in resilient wheels of railway vehicles. The examined naturally aged segments have different service history. The effect of aging on the response to dynamical loading was investigated. Specimens sliced from segments were subjected to dynamic mechanical analysis which consists in sinusoidal compressive strain controlled loading with different amplitudes and frequencies. The phase angle δ between strain and stress responses and storage and loss moduli were determined by different evaluation methods. Rubber becomes harder in course of aging, loses its damping capability, properties affecting its friction behavior change and friction coefficient is reduced in comparison with the virgin state.

1. Introduction

In the urban transport (suburban trains, metros, light rail vehicles, tramcars) comfort, noise-damping and protection of wheels, wear resistance and performance under braking are permanently in an agenda of researchers. Isoprene/Butadiene rubber segments used in resilient wheels effectively reduce vibrations and noise in transportation. Beside their damping function, the rubber segments have to transmit considerable alternating shear force and to sustain a large cyclic compressive load superposed on large static pre-strain. Their material is degraded owing to severe operational conditions such as heavy mechanical loading, thermal loading due to the internal dissipative heating and to external temperature variation. In addition, permeation of oxidative, photolytic and hydrolytic agents causes deterioration in the material properties particularly of the outside surface. Rubber ages and becomes harder; it loses its damping capability and those properties determining its friction behaviour change. A long-lasting compressive load changes the segment shape and dimensions and produces a permanent set. After a certain period, slips between the wheel and rubber segments occur and the segments fail to perform their function without any apparent damage. The aging effects on the dynamical and tribological properties of naturally aged segments with different service history were experimentally investigated. Estimates of service life can then be made by extrapolating the degree of degradation after a given time of functioning.

Samples sliced by water jet from segments were subjected to a series of static and relaxation tests, to dynamic mechanical analysis, as well as to hardness and friction testing.

The experimental measurement of the time dependent response and of damping properties of viscoelastic materials consists of performing creep and stress relaxation tests which are suitable for studying the material response over a long period of time.

We previously investigated the quasistatic rate-dependent behaviour of virgin segments of isoprene-butadiene rubber in compression regimes. The behaviour at different strain levels

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was examined in detail through quasistatic cyclic tests and in simple and multistep relaxation tests. The viscosity-induced rate-dependent effects were described and parameters of the material model were determined. The model was implemented into FEcode [1]. Tribological properties of segment rubber were also investigated [2]. The present paper is focused on the dynamic mechanical analysis of virgin and aged segments.

The dynamic mechanical analysis (DMA) is well suited for the identification of the short-time range of polymer response. DMA consists of dynamic tests, in which the force resulting from a sinusoidal strain controlled loading is measured.

The dynamic behaviour of filler-reinforced rubber has been investigated by many material scientists. The dependence of the storage and dissipation modulus on the temperature, the predeformation, the deformation amplitude and the frequency were all investigated. It was demonstrated that the moduli also depend on the type of filler material. Payne [3] first pointed out that the moduli of carbon black filled rubber lessen with increasing deformation amplitudes. By means of further tests he reached the conclusion that this behaviour must be attributed to a thixotropic change. Lion [4] observed that both the storage and the dissipation modulus depend on the frequency of the deformation process. This variation is weakly pronounced and in a good approximation of power-law type. In terms of the theory of linear viscoelasticity, this behaviour corresponds to a continuous relaxation time distribution. With increasing temperatures, he observed both a decrease in moduli and a lessening of the frequency dependence. The dependence of the dynamic moduli on the filler content and the static predeformation has been investigated in detail by [5]. When a viscoelastic material is subjected to a sinusoidally varying strain, the stationary stress-response will be reached after some initial transients and the resulting stress is also sinusoidal, having the same angular frequency but advanced in phase by an angle δ. Then the strain lags the stress by the phase angle δ. The axial displacement *u(t)* consists of a static predeformation u_0 under compression which is superimposed by small sinusoidal oscillations:

$$u(t) = u_0 + \Delta u \, sin(2\pi ft). \tag{1}$$

Stresses and strains are calculated with respect to the reference geometry of the pre-deformed specimen:

$$\varepsilon_0 = u_0 / (L_0 + u_0), \quad \Delta\varepsilon = \Delta u / (L_0 + u_0), \tag{2}$$

where L_0 is the undeformed length of the specimen. The force response *F(t)* of the specimen is a harmonic function and can be written as :

$$F(t) = F_0 + \Delta F \sin(2\pi ft + \delta). \tag{3}$$

F_0 is the static force depending only on the pre-deformation u_0. The force amplitude ΔF and the phase angle δ depends, in general, on the pre-deformation, the frequency and the strain amplitude ([6], [7]). If the incompressibility of the rubber is assumed $A_0L_0=A(L_0+u_0)$, where A_0 is the cross-sectional area of the undeformed specimen, we can relate the force to the cross-sectional area A of the pre-deformed specimen:

$$\begin{aligned} \sigma(t) = \frac{F(t)}{A} = \sigma_0 + \\ +\Delta\sigma\left[cos(\delta)sin(2\pi ft) + sin(\delta)cos(2\pi ft)\right]. \end{aligned} \tag{4}$$

The dynamic stress-response $\sigma(t)$ normalised by the deformation amplitude $\Delta\varepsilon$ can be written:

$$\begin{aligned}\sigma(t)=\sigma_0+\Delta\varepsilon[G'(\varepsilon_0,f,\Delta\varepsilon)sin(2\pi ft)+\\+G''(\varepsilon_0,f,\Delta\varepsilon)cos(2\pi ft)],\end{aligned} \tag{5}$$

where

$$G'(\varepsilon_0,f,\Delta\varepsilon)=\frac{\Delta\sigma}{\Delta\varepsilon}cos(\delta), \tag{6a}$$

and

$$G''(\varepsilon_0,f,\Delta\varepsilon)=\frac{\Delta\sigma}{\Delta\varepsilon}sin(\delta) \tag{6b}$$

are the storage and dissipation moduli respectively, and δ is the phase angle. In general, carbon black-reinforced rubber has fairly a weak frequency dependence in conjunction with a pronounced amplitude dependence [7]. If the strain amplitude $\Delta\varepsilon$ increases, the storage modulus G' lessens and the loss modulus G" shows a more or less pronounced sigmoidal behaviour - Payne effect. If the material is linear viscoelastic, then these two moduli depend neither on the deformation amplitude nor on the static predeformation. The damping factor or loss tangent (tan δ) which is the ratio G"/ G' is the measure of mechanical energy dissipated as heat during the dynamic cycle. If the dynamic strain amplitude is constant in time, we can observe time-independent moduli [8]. These phenomena are frequently interpreted as a dynamic state of equilibrium between breakage and recovery of physical bonds linking adjacent filler clusters. The most common model of this state is the Kraus model ([9], [10]) which describes the amplitude dependence of dynamic moduli. The influence of static pre-deformation ε_0 is included in the models of [11], [12] and the uniaxial form of the frequency, amplitude and pre-strain dependent on dynamical modulus is proposed by [8].

The purpose of this present paper is to summarise the results of experimental research into the behaviour of rubber samples with different service history under dynamic loading conditions. We carried out harmonic strain-controlled tests under compression and studied the dependence of the storage and dissipation moduli on the frequency, on the deformation amplitude and on the static pre-strain.

2. Experimental

The sample is a rectangular parallelepiped whose width, depth, and height are 15, 15, and 20 mm respectively. The specimens were cut by water jet from segments that have been in operation for different times. Since the operating conditions are not known, the only measure of segment fatigue is the number of kilometres travelled. This number, in thousands of kilometres, is shown in Table 1, together with the hardness values. Specimens marked R are all from one producer, the specimen marked G is of unknown provenance.

The tests are performed at room temperature using an electrodynamic Instron testing machine. In the preconditioning process applied, the pre-strain was about λ_0=0.65 under compression and the amplitude $\Delta\varepsilon$ about 0.08.

Table 1: *Hardness of samples and the path in thousands of km.*

Sample	0 R	1 R	2 R	3 G	4 R	5 R	6 R	Sample
[km]	0	175	255	172	75	473	146	[km]
ShoreA	70.3	82.4	86.1	85.4	83.6	83.5	88.3	ShoreA

Subsequent to this preconditioning process, three different constant compressive pre-strains λ_0=0.85, 0.75 and 0.65 were applied, and for each of them, five ascending amplitudes of the superimposed harmonic strain varied between 0.01 and 0.06. The frequencies varied in 5 steps between 1 and 10 Hz for each strain amplitude. The process was strain-controlled. Raw test data sampled at 1 kHz were recorded by PC.

In majority specialized DMA testing devices the command signal generation and sequencing, data acquisition and subsequent data processing are accomplished using the inherent apparatus software. Such equipment was not at our disposal therefore we processed recorded signals ourselves by the sequence of different evaluating methods.

In order to determine the storage and loss moduli of the material, the static predeformation u_0, the amplitude Δu and the frequency f of strain controlled loading, corresponding parameters of the force response and the phase angle δ must be extracted from the recorded raw signals. We supposed that the raw signals, i.e. head displacement u(t) and force F(t), are given approximately by the harmonic functions (1) and (3) and we used two numerical methods for their processing:

1) Discrete Fourier transform (DFT): The DFT was used first in order to determine the main frequency and the phase shift of the signals *u(t)* and *F(t)*. For this purpose the magnitudes of signals were shifted by their mean values which were supposed to be approximately the static values u_0 and F_0 and the function fft of Matlab was applied. DFT verified that the main frequencies of both signals do not differ from the supposed excitation frequency. The phase shift between the force and displacement signals was determined as the difference between the phases of both signals at the excitation frequency.

2) Virtual vector-voltmeter method (VV): The VV method is suitable for the determination of the phase shift between two signals with the same known frequency f. This method works like the narrow-band filtration and is suited for noisy signals [13]. The recorded signal is first multiplied by the reference sinusoidal signal and then by the reference signal shifted by π/2. For example the phase shift between the measured displacement and the reference signals is calculated:

$$\varphi_{uR} = arctg \frac{\sum\limits_{(n)} \left[\Delta u_n \ sin\left(2\pi f t_n + \pi/2\right) \right]}{\sum\limits_{(n)} \left[\Delta u_n \ sin\left(2\pi f t_n\right) \right]}, \tag{7}$$

where

$$\Delta u_n = u_n - \frac{1}{n} \sum_{(n)} u_n \ .$$

The phase shift between the force and displacement signals is calculated as the difference:

$$\delta = \varphi_{FR} - \varphi_{uR}. \tag{8}$$

For the numerical computation the Signal Processing Toolbox and the Optimization Toolbox of Matlab were used. All methods were applied to the same integer number of signal periods. The frequency and the phase shift δ between the signals were determined by the DFT method. The VV method is suitable for the phase angle evaluation. The discrete Fourier transform was used to determine the frequency content of force and head displacement signals and to calculate the phase lag between them. Furthermore, we determined the complex dynamic modulus as the ratio between the amplitudes of stress and strain and dynamic moduli were calculated according to the relation (6).

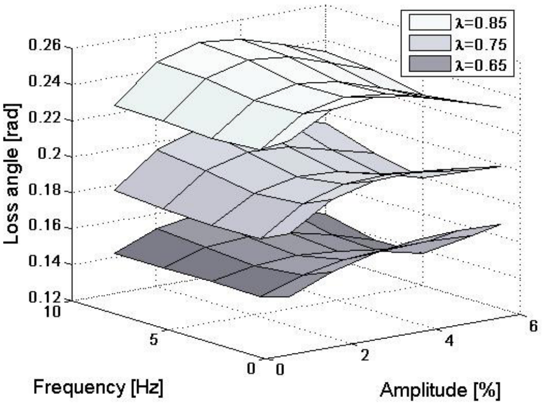


Figure 1: Variation of loss angle of virgin specimens with amplitude and frequency at different pre-strains λ.

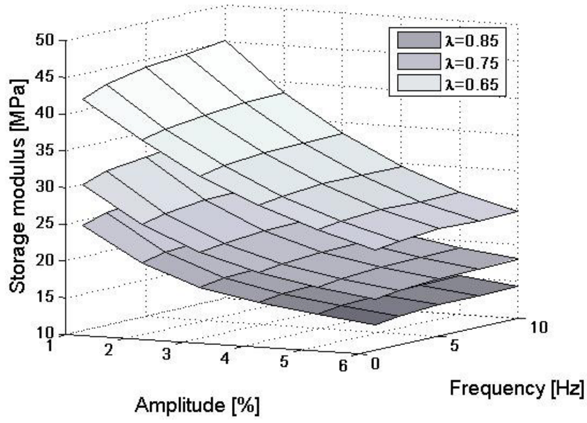


Figure 2: Variation of storage modulus of virgin specimens with amplitude and frequency at different pre-strains λ.

3. Results of Measurement

3.1. Dynamic properties of virgin samples

The influence of the static pre-deformation (λ_0 increasing from 0.85 over 0.75 to 0.65) on the loss angle and dynamic moduli was monitored for the virgin samples [14-16].

The evolution of loss angle δ of virgin material for the three compressive pre-deformations is evident from the Figure 1. The loss angle decreases markedly with increasing pre-strain and the storage modulus increases as is evident from the Figure 2.

In the measured range of amplitudes and frequencies the graph of loss angle resembles a horse saddle. The frequency dependence of loss angle δ is weak; it passes through a moderate minimum between 1 and 3 Hz. By contrast, the amplitude dependency is pronounced; for each experimental frequency the loss angle reaches its maximum approximately in the middle of the interval of amplitude.

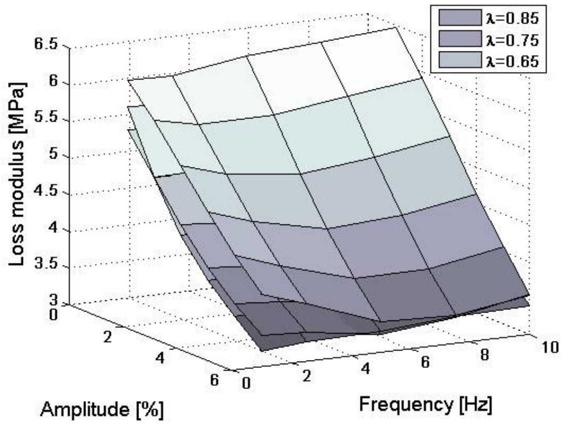
The storage modulus of virgin specimens on Figure 2 increases notably with increasing static pre-stretch λ_0. The storage modulus increases slightly with increasing frequency and significantly decreases with increasing amplitude. Both dependencies are monotonous in the range of frequencies and amplitudes applied.

The loss modulus of virgin specimens on the Figure 3 follows similar trends with the difference that it does not depend significantly on the static pre-stretch λ_0.

3.2. Dynamic properties of aged samples

The main task of this experimental work was to investigate how the natural aging and fatigue affect mechanical properties of rubber segments.

The dynamic properties were measured by means of DMA on 6 sets of aged samples. In each set, there were 5 samples with the same operational history which is evident from Table 1.



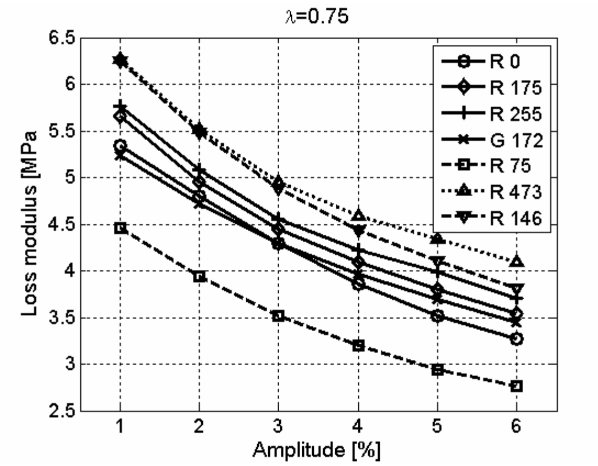


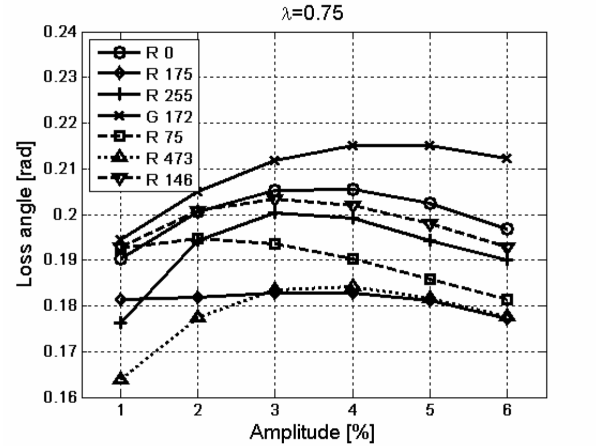
Figure 3: Variation of loss modulus of virgin specimens with amplitude and frequency at different pre-strains λ.

Figure 4: Comparison of loss moduli of specimens with different service history at λ_0=0.75 and at frequency 5 Hz.

3.2.1. Loss angle δ

Comparison of loss angles of samples with a different service history is on Figure 5. The graph represents the dependency on amplitudes at the static pre-strain λ_0=0.75 and the frequency 5 Hz.

We see that the loss angles of aged and virgin samples have a similar trend. The loss angle values of aged specimens are lower than the loss angle of virgin specimens. The only exception is the set of samples G172, which is from a different producer.



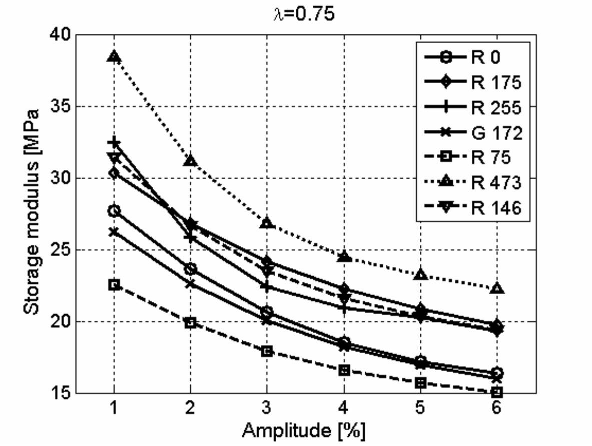


Figure 5: Comparison of loss angles of specimens with different service history at λ_0=0.75 and at frequency 5 Hz.

Figure 6: Comparison of storage moduli of specimens with different service history at λ_0=0.75 and at frequency 5 Hz.

3.2.2. Storage modulus

Comparison of storage moduli of specimens with different service history is on Figure 6. We see a considerable fall of storage moduli of all samples with increasing values of amplitude. An analysis of the measured data shows that the dependence of storage moduli on frequency is weak for all samples in the applied frequency interval.

The majority of the storage moduli of aged samples are bigger that the storage modulus of virgin material. The sample R473 that has been in the longest operation shows the largest storage modulus.

3.2.3. Loss modulus

A comparison of loss moduli of aged and virgin samples is on Figure 4. Loss moduli diminish with increasing amplitude. The difference of their values is small, nevertheless, the loss modulus of virgin material is lower than most of the other values.

3.3. Hardness of segments and permanent set

The permanent set and the hardness of all aged segments were measured prior to being sliced into samples by water jet.

3.3.1. Permanent set

The average thickness reducing permanent set in the direction of main compressive working load is about 11.5 %. The extending permanent set is about 16.2 % in the circumferential direction of the wheel. In this direction, the segments are subjected to shear due to the torque transmission, and they are also enlarged owing to the compression in a perpendicular direction.

3.3.2. Hardness

Hardness Shore A of rubber segments is listed in the Table 1. The hardness on the surface of the worn segments is 10-15 ShA more than hardness of virgin segments. The reason for such a difference is probably the enhanced degradation of surface layers.

4. Results and conclusion

DMA tests were conducted under strain-controlled loading condition at room temperature to prove the assumed difference of dynamical properties amongst IR/BR samples prepared from naturally aged segments with a different service history. Based on the test results we can draw some conclusions:
All samples show the Payne effect – as the strain amplitudes of the load increase, storage and loss moduli decrease.

- The frequency dependence of observed dynamic quantities is weak in the experimental frequency range.
- The loss angle decreases and the storage modulus increases with the increasing static pre-strain.
- The loss angles of virgin rubber samples are larger than of worn ones.
- Storage moduli of samples from worn segments are greater than storage moduli of virgin samples.
- The segments become harder and stiffer during their service life and lose their damping capability.
- Dimensions of segment change markedly due to the permanent set.

For all these reasons segments can fail to perform their function which is quite unusual for a rubber element. In addition to the vibration damping, they have to transmit the driving torque by means of friction. Usually the slip between the wheel disc and rim put an end to segment life. The main purpose of this study was to examine this one aspect of the segment failing. An exhaustive study of segment reliability remains a future endeavour.

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