DYNAMIC TESTING PROCEDURE:
NEW DOCUMENT

Testing Procedure for Measurement of Dynamic Properties of Vulcanized Rubber

INSTITUTION OF THE RUBBER INDUSTRY

RUBBER IN ENGINEERING DESIGN GROUP.

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In November 1971 the Rubber in Engineering Design Group, under the then Chairman P. B. Lindley, decided that there was need for a standardized test procedure for measuring the dynamic properties of vulcanized rubbers of the types used in engineering applications. Accordingly a working party was formed, with A. G. Thomas as Chairman and A. B. Worthington as Secretary, to look into the question and to see what documents could usefully be produced. The membership of the working party was carefully chosen so that there was good representation from both the rubber manufacturers and the users. The latter included the car manufacturers, British Rail, consulting engineers interested especially in civil engineering, and the Ministry of Defence. The object of having such a wide range of interests represented was to ensure that any recommendations should produce data which would both satisfy the users' requirements and also be realistic for manufacturers to provide. Although it is obviously necessary for the behaviour of the final engineering component to be measured, the wide variety of such components and their correspondingly different characteristics made it very difficult to formulate any generalized useful testing procedures for them. It was therefore decided initially to concentrate on defining a test procedure for evaluating the materials themselves. The outcome of the work of the working party is this document.

It was presented to the relevant committee at the Cologne ISO meeting in September 1972, and met with general approval. It appears probable that an ISO draft standard which is being prepared will be consistent with the general approach given here. A British Standard based on this document is in an early draft form.

The main reason for publishing the document here is to invite comments from an even wider audience than that covered by the working party membership. Such comments should be sent to A. B. Worthington, RAPRA, Shawbury, Shrewsbury, SY4 4NR.

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Testing Procedure for Measurement of Dynamic Properties of Vulcanized Rubber

1. Scope

The purpose of this recommendation is to give guidance for the testing of rubber vulcanizates under dynamic conditions so that the viscoelastic properties of importance in engineering design can be determined. The emphasis is therefore on testing material properties under conditions relevant to engineering applications. Considerations of creep, stress relaxation and durability are excluded.

2. Description of Terms

The following terms are strictly applicable only to a linear material, that is, one for which the stress is proportional to the deformation. For materials containing substantial quantities of filler this is not true, and the terms cannot therefore have a precise significance. However, if the amplitude of deformation is held constant, effective values of these quantities can be measured on the understanding that their magnitude depends on the deformation amplitude.

Storage modulus (*in-phase modulus*) G'. The component of applied shear stress which is in phase with the shear strain, divided by the shear strain.

Loss modulus (*out-of-phase modulus*) G''. The component of applied shear stress which is 90° out of phase with the shear strain, divided by the shear strain.

Complex modulus $G^* = G' + jG''$. The resultant shear stress divided by the resultant shear strain where each is a vector which may be represented by a complex number.

Absolute value of the complex modulus $|G^*| = (G'^2 + G''^2)^{\frac{1}{2}}$. The magnitude of the complex modulus.

Loss angle δ. The phase angle between the sinusoidal force and displacement.

Loss factor (*loss tangent*). Tan δ is given by

$$\tan\delta = \frac{G''}{G'}$$

The following terms, although not appearing in the body of this document, are frequently used in other test procedures and in design calculations.

Logarithmic decrement Λ. The natural logarithm of the ratio between successive amplitudes of the same sign in a damped free oscillation. In certain cases, e.g. for the free torsional oscillation of a rubber cylinder of circular cross section connected to an inertia bar, it is approximately related to the loss factor by

$$\tan\delta = \frac{\Lambda}{\pi\left(1 + \frac{\Lambda^2}{4\pi^2}\right)}$$

Damping Ratio u. The ratio between actual and critical damping where critical damping is that required for the borderline condition between oscillatory and non-oscillatory behaviour. Damping ratio is given in terms of the logarithmic decrement thus:

$$u = \frac{\Lambda}{2\pi\sqrt{1 + \frac{\Lambda^2}{4\pi^2}}} = \sin\arctan\left(\frac{\Lambda}{2\pi}\right)$$

$$\simeq \tfrac{1}{2}\tan\delta \text{ for small values of } \delta$$

Rebound Resilience R. The ratio between the output and input energies of a moving mass which impacts a test piece. It is approximately related to the loss factor thus:

$$R = e^{-\pi\tan\delta}$$

This approximation is not very accurate, partly because of the dependence of tan δ on strain, the latter not being controlled in a resilience measurement. Tan δ must be measured at a frequency given by $\frac{1}{2\tau}$ where τ is the duration of impact in the resilience measurement.

Peak Transmissibility Q. The value of the transmissibility at resonance of a system consisting of a mass supported by a rubber spring. It is given by

$$Q = \frac{(1 + \tan^2\delta)^{\frac{1}{2}}}{\tan\delta}$$

$$\simeq \frac{1}{\tan\delta} \text{ for small values of } \delta$$

3. Type of Deformation

The deformation used shall be simple shear. This deformation has the merits that (a) a substantial proportion of manufactured articles are used in this type of strain, (b) the stress-strain behaviour is more nearly linear than in tension or compression, especially for compounds containing little filler. Sinusoidal forced oscillations of a constant frequency and strain amplitude shall be employed as the method of measurement. Forced oscillations, rather than resonance or free oscillation methods, are used because this ensures control of the strain amplitude, which, as discussed in Section 7, is important.

4. Test piece

The test piece shall be of either circular or square cross section and bonded to rigid end plates. Bonding during vulcanization is to be preferred, as this produces the most reliable bond. Sticking vulcanized rubber adequately to metal is not easy, although with care satisfactory results can be obtained.

To avoid significant bending, the diameter (or side in the case of square test pieces) shall be at least four times the thickness[1]. This will ensure that the deformation is essentially simple shear of the calculated magnitude and that the apparent shear modulus differs by less than three per cent from the true value.

It is not proposed to make recommendations for absolute test piece dimensions because of the range of sensitivities of the testing machines available. However, the difficulties of ensuring uniform vulcanization in thick samples suggest that thicknesses of greater than 12 mm are best avoided.

Double shear test pieces of the general form shown in Fig 1 are preferred as being the most convenient. The measured properties will, of course, be an average of the two samples.

5. Test machine

Any test machine whether mechanical, hydraulic or electromagnetic may be used provided the displacements and forces are adequate to give the forced oscillations of the necessary amplitudes over the required frequency range. The machine shall be such that the results can be obtained with the precision required in section 10.

6. Temperature of test

The temperature of test may be chosen for the particular

Appendix I Temperature Rise on Cycling

The rate of energy loss per unit volume per cycle is given by $\pi.\ \sin\delta.\ |G^*|.\ e^2$ where $|G^*|$ is the absolute value of the complex modulus of elasticity, given by $|G^*| = (G'^2 + G''^2)^{\frac{1}{2}}$, and e is the strain amplitude. Thus the rate of rise of temperature when there are no heat losses from the specimen is

$$\frac{\pi \sin\delta\,|G^*|\; e^2 \text{ x (frequency of cycling)}}{\text{Heat capacity per unit volume}}\ .$$

The heat capacity must be in the appropriate units, viz. Joules per cubic metre.

For a strain amplitude of ± 10% ($e = 0{\cdot}1$), $|G^*| = 1500$ kN/m², $\delta = 10°$, a heat capacity of $1{\cdot}7 \times 10^6$ j/m³ ($\simeq 0{\cdot}4$ cals/cm³) and a frequency of cycling of 15 Hz, the rate of rise of temperature would be 0·072 °C/s. This is for a poorly resilient material of about 70 IRHD. To ensure that the temperature rise does not exceed about 2 °C, which may introduce as much as a two per cent error in the stiffness, the time of test should therefore be kept below about 30 s for such a material.

Most materials used in engineering applications have loss angles of significantly less than 10°, so that the temperature rise will present less of a problem.

Appendix II Calculation of Dynamic Properties from the Force-Deflection Loop

Fig 2 shows a force-deflection loop obtained from a dynamic test. The origin O represents the mean values of the force and deflection, and, if a static deflection is imposed, will not be the zero values. The forces and deflections shown are thus the dynamic components.

If the behaviour of the rubber is linear, that is, a deflection varying sinusoidally with time gives an accurately sinusoidal force variation, the loop shown in Fig 2 will be an ellipse. In this case, the absolute value of the complex modulus is given by

$$|G^*| = \frac{f_o h}{A x_o} \qquad (1)$$

where f_o and x_o are the maximum force and deflection amplitudes respectively, A is the effective cross sectional area and h, the thickness of the test piece. Thus $\frac{f_o}{x_o}$ is given (see Fig 2) by the slope of the line OA which is the diagonal of the circumscribed rectangle.

The loss angle δ is given by

$$\tan\delta = \frac{f_2}{f_1} \qquad (2)$$

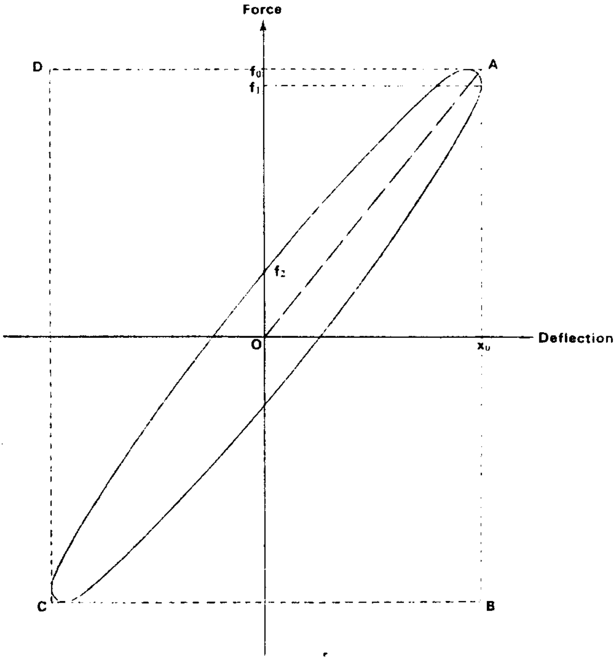

Fig. 2.

The storage (or in-phase) modulus G' is given by

$$G' = \frac{f_1 h}{A x_o} \qquad (3)$$

and the loss modulus G″, by

$$G'' = \frac{f_2 h}{A x_o} \qquad (4)$$

The loss angle is also given by

$$\sin\delta = \frac{\text{(length x breadth of ellipse)}}{\text{(area of circumscribing rectangle ABCD)}} \qquad (5)$$

$$= \frac{4 \text{ x (area of ellipse)}}{\pi \text{ x (area ABCD)}}$$

$$= \frac{\text{area of ellipse}}{\pi f_o x_o} \qquad (6)$$

This latter relation may be particularly useful when there is some non-linearity and the ellipse is not perfect, as it will give an average δ value.

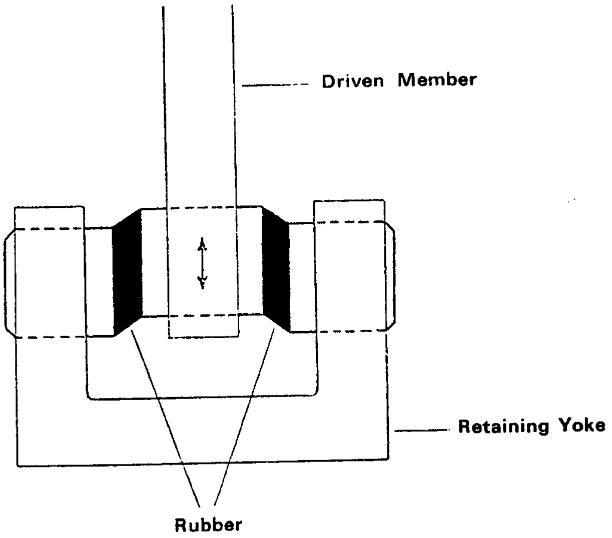

Fig. 1. Schematic arrangement of test-piece and mounting. The displacement of the driven member and the force experienced by the yoke are monitored by suitable transducers.

application in mind, but it is preferred that it be one of the following temperatures for the purpose of standardization: -55, -40, -25, 0, +20, +50, +70, +100, +125, +150, +175, +200 °C. Care must be taken to ensure that the test piece has reached the required temperature.

The properties of rubbers, particularly those containing fillers, are sensitive to temperature. For example, a change of 1 °C produces about a one per cent change in modulus for a typical filled rubber at 20 °C. Thus, to avoid errors due to temperature variation, it is necessary that the temperature be controlled to ± 1 °C.

7. Frequency and Strain Amplitude

It is well known that materials containing substantial quantities of filler show viscoelastic behaviour that is dependent on the strain amplitude of test and on the strain history of the test piece. It is thus in general necessary to control these factors. Unfilled materials do not have these complications to a significant extent.

A difficulty that can arise in testing materials of high loss angle is that heat generation in the sample may raise the temperature significantly during the test and, as the properties of filled vulcanizates depend quite markedly on temperature, this introduces errors. This point is dealt with in Appendix I. Considerations of this factor, and the strains and frequencies met with in practical applications, have led to the recommendations that up to a frequency of 15 Hz, the strain amplitudes of test shall be ± 2% and ± 10%.

Observations at these two strains will determine the magnitude of any strain amplitude effect that may be present. It may then be desirable to augment the observations by tests at other strains if the application in mind demands this. For larger strains the possible temperature rise in the sample must however be borne in mind.

Above 15 Hz the amplitudes sustained in practice decrease with increasing frequency approximately as $(\text{frequency})^{-2}$, that is, the maximum acceleration remains approximately constant. It is therefore recommended that the test strain amplitude decreases from ± 10% according to this relation for frequencies above 15 Hz and will thus be as given in the following table:—

Table 1

Frequency Hz	0·1	1	10	15	30	50	100	200
Strain Amplitude %	10	10	10	10	2·5	0·9	0·225	0·056

As discussed previously, at frequencies of 15 Hz and below tests shall also be carried out at strain amplitudes of ± 2% so that any effect of amplitude can be detected.

The actual frequencies may be chosen for relevance to the particular application in mind, but it is preferred that they be from the above list for purposes of standardization.

8. Superposed Static Strains

In practice, many articles are used in a combination of strains, such as combined compression and shear. Because of the wide variety of combinations possible in practice it is not proposed to recommend precise values of static strains. However, the recommendations given here can be applied to the dynamic shear component whether or not a static strain is present.

9. General Test Procedure

The test piece should be rested unstrained for at least 24 hours after vulcanization.

The testing up to 15 Hz shall be carried out first at the lower strain of ± 2%, and the strain then increased to ± 10%. Because of the hazard of temperature rise, which may be significant at the higher strain at 15 Hz (see Appendix I), the period of oscillation at this strain should be kept to the minimum consistent with obtaining accurate results. A maximum desirable time of 30 s is suggested by the calculation in Appendix I, although this obviously depends on the dynamic properties of the rubber. If time-dependent changes occur during the test which can be ascribed to a temperature rise, the results should be extrapolated to zero time to get the dynamic properties appropriate to the nominal test temperature. At higher frequencies the test amplitude should be lower, as given in Section 7. Temperature rise at these higher frequencies is unlikely to be a problem at the amplitudes recommended. It should be noted that the modulus at low deformations is depressed by prior oscillations at higher deformations, some hours of resting being necessary for the effect to disappear[2]. Thus, if tests above 15 Hz are required, it is desirable to begin at the higher frequencies (and thus smaller amplitudes) and reduce the frequency (increasing the amplitude), so avoiding this possible complication.

10. Presentation of results

It is recommended that the results be presented as the variation of tan δ and $|G^*|$ with temperature, frequency and amplitude. The attainable accuracy in tan δ should be of the order of ± 0·01, or ± 5% whichever is the greater, and ± 2% in $|G^*|$. This is considered to be adequate accuracy for the purposes for which the results are likely to be put in engineering applications.

Appendix II indicates how these quantities can be determined from a force-deflection curve. If both the force and displacement are sinusoidal with respect to time, the signals from the transducers can be analysed by suitable electronic techniques to give the required information without recourse to recording the force-deflection loop. However, filled rubbers may exhibit some non-linearity in behaviour which complicates this treatment, and measurement of the area of the force-deflection loop as described in Appendix II may be the most realistic procedure. In this case the accuracy attainable may be only about one half of that given above.

concluded over

REFERENCES

1. Rivlin, R. S. and Saunders, D. W., Trans IRI 24, 296 (1949).
2. Fletcher, W. P. and Gent, A. N., Trans IRI 26, 45 (1950).