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Mechanical vibration and shock — Characterization of the dynamic mechanical properties of visco-elastic materials —

Part 3: Cantilever shear beam method

Vibrations et chocs mécaniques — Caractérisation des propriétés mécaniques dynamiques des matériaux visco-élastiques —

Partie 3: Méthode du faisceau par cisaillement en encorbellement

Reference number
ISO 18437-3:2005(E)

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18437-3 was prepared by Technical Committee ISO/TC 108, *Mechanical vibration and shock*.

ISO 18437 consists of the following parts, under the general title *Mechanical vibration and shock — Characterization of the dynamic mechanical properties of visco-elastic materials*:

— *Part 2: Resonance method*

— *Part 3: Cantilever shear beam method*

Part 4 (*Impedance method*) is under preparation.

Introduction

Visco-elastic materials are used extensively to reduce vibration magnitudes in structural systems through the dissipation of energy (damping) or isolation of components, and in acoustical applications that require a modification of the reflection, transmission, or absorption of energy. Such systems often require specific dynamic mechanical properties in order to function in an optimum manner. Energy dissipation is due to interactions on the molecular scale and is measured in terms of the lag between stress and strain in the material. The visco-elastic properties (modulus and loss factor) of most materials depend on frequency, temperature and strain magnitude. The choice of a specific material for a given application determines the system performance. The goal of this part of ISO 18437 is to provide details on the principle of operation of a cantilever shear beam method that avoids common clamping errors through the use of fixed ends, the measurement equipment, in performing the measurements, and analysing the resultant data. A further intent is to assist users of this method and to provide uniformity in the use of this method. This part of ISO 18437 applies to the linear behaviour observed at small strain magnitudes.

Mechanical vibration and shock — Characterization of the dynamic mechanical properties of visco-elastic materials —

Part 3:
Cantilever shear beam method

1 Scope

This part of ISO 18437 defines a cantilever shear beam method for determining from laboratory measurements the dynamic mechanical properties of the resilient materials used in vibration isolators. Common errors due to clamping the specimen are avoided by using fixed ends so there is no rotational motion of the beam at its ends. This part of ISO 18437 is applicable to shock and vibration systems operating from a fraction of a hertz to about 20 kHz.

This part of ISO 18437 is applicable to resilient materials that are used in vibration isolators in order to reduce

a) transmissions of unwanted vibrations from machines, structures or vehicles that radiate sound (fluid-borne, airborne, structure-borne, or others), and

b) the transmission of low-frequency vibrations that act upon humans or cause damage to structures or sensitive equipment when the vibration is too severe.

The data obtained with the measurement methods that are outlined in this part of ISO 18437 and further detailed in ISO 18437-2 are used for

— the design of efficient vibration isolators,

— the selection of an optimum material for a given design,

— the theoretical computation of the transfer of vibrations through isolators,

— information during product development,

— product information provided by manufacturers and suppliers, and

— quality control.

The condition for the validity of the measurement method is linearity of the vibrational behaviour of the isolator. This includes elastic elements with nonlinear static load deflection characteristics, provided that the elements show approximate linearity in their vibrational behaviour for a given static preload.

Measurements using this method are made over two decades in frequency (typically 0,3 Hz to 30 Hz) at a number of temperatures. By applying the time-temperature superposition principle, the measured data are shifted to generate dynamic mechanical properties over a much wider range of frequencies (typically 10^{-3} Hz to 10^{9} Hz at a single reference temperature) than initially measured at a given temperature.

NOTE For the purpose of this part of ISO 18437, the term “dynamic mechanical properties” refers to the determination of the fundamental elastic properties, e.g. the complex Young's modulus as a function of temperature and frequency and, if applicable, a static preload.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472:1999, *Plastics — Vocabulary*

ISO 2041:1990, *Vibration and shock — Vocabulary*

ISO 4664-1:2005, *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 1: General guidance*

ISO 6721-1:2001, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

ISO 10112:1991, *Damping materials — Graphical presentation of the complex modulus*

ISO 10846-1:1997, *Acoustics and vibration — Laboratory measurement of vibro-acoustic transfer properties of resilient elements — Part 1: Principles and guidelines*

ISO 23529:2004, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472, ISO 2041, ISO 4664-1, ISO 6721-1, ISO 10112, ISO 10846-1, ISO 23529 and following terms and definitions apply.

3.1
Young's modulus
E^*
quotient of normal stress (tensile or compressive) to resulting normal strain, or fractional change in length

NOTE 1 Unit is the pascal (Pa).

NOTE 2 Young's modulus for visco-elastic materials is a complex quantity, having a real part E' and an imaginary part E''.

NOTE 3 Physically, the real component of Young's modulus represents elastic-stored mechanical energy. The imaginary component is a measure of mechanical energy loss. See 3.2.

3.2
loss factor
ratio of the imaginary part of the Young's modulus of a material to the real part of the Young's modulus (the tangent of the argument of the complex Young's modulus)

NOTE When there is energy loss in a material, the strain lags the stress by a phase angle, δ. The loss factor is equal to tan δ.

3.3
time-temperature superposition
principle by which, for visco-elastic materials, time and temperature are equivalent to the extent that data at one temperature are superimposed upon data taken at a different temperature merely by shifting the data curves along the frequency axis

3.4
shift factor
measure of the amount of shift along the logarithmic (base 10) axis of frequency for one set of constant-temperature data to superpose upon another set of data

3.5
glass transition temperature
T_g
temperature at which a visco-elastic material changes state from glassy to rubbery, and corresponds to a change in slope in a plot of specific volume against temperature

NOTE 1 Unit is degrees Celsius (°C).

NOTE 2 The glass transition temperature is typically determined from the inflection point of a specific heat vs. temperature plot and represents an intrinsic material property.

NOTE 3 T_g is not the peak in the dynamic mechanical loss factor. That peak occurs at a higher temperature than T_g and varies with the measurement frequency; hence is not an intrinsic material property.

3.6
resilient material
visco-elastic material intended to reduce the transmission of vibration, shock or noise

NOTE 1 It is sometimes referred to as an elastic support, vibration isolator, shock mounting, absorber or decoupler.

NOTE 2 The reduction may be accomplished by the material working in tension, compression, torsion, shear, or a combination of these.

3.7
linearity
property of the dynamic behaviour of a resilient material if it satisfies the principle of superposition

NOTE 1 The principle of superposition is stated as follows: if an input $x_1(t)$ produces an output $y_1(t)$ and in a separate test an input $x_2(t)$ produces an output $y_2(t)$, superposition holds if the input $\alpha x_1(t) + \beta x_2(t)$ produces the output $\alpha y_1(t) + \beta y_2(t)$. This holds for all values of α, β and $x_1(t)$, $x_2(t)$, where α and β are arbitrary constants.

NOTE 2 In practice, the above test for linearity is impractical. Measuring the dynamic modulus for a range of input levels can provide a limited check of linearity. For a specific preload, if the dynamic transfer modulus is nominally invariant, the system measurement is considered linear. In effect this procedure checks for a proportional relationship between the response and the excitation.

4 Test equipment (see Figure 1)

4.1 Electro-dynamic vibration generator

The vibration generator induces an oscillating sinusoidal cantilever shear strain into the sample beam at the selected frequency. An electro-dynamic vibration generator, with the following specifications, is typical of that required to provide a driving force for the specimen in a typical test:

— frequency range: 0,3 Hz to 30 Hz;

— force rating: $>$ 10 N;

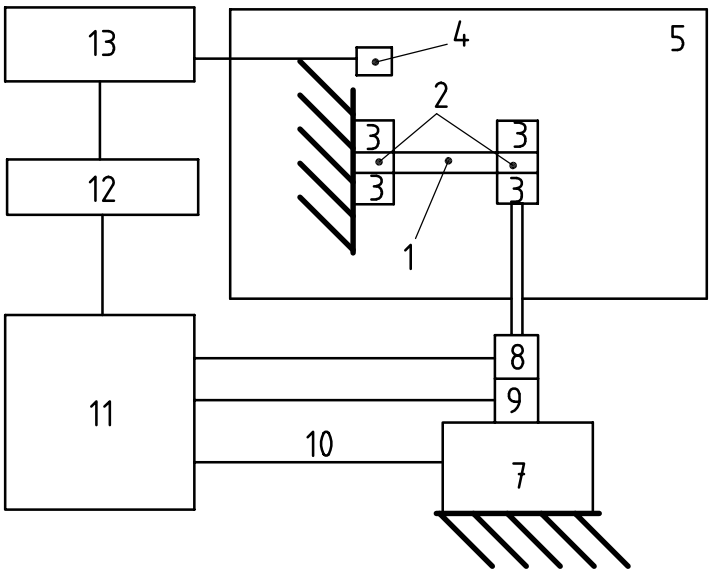
— amplitude: $\approx$ 100 μm.

4.2 Force measurement

Typically the force is inferred by measuring the magnitude and phase of the current driving the electro-dynamic vibration generator. The force shall be calibrated using a known mass. The following specifications apply:

— frequency range: 0,3 Hz to 30 Hz;

— uncertainty: $<$ 0,5 %.



Key

1 beam specimen
2 specimen end blocks
3 specimen clamps
4 temperature probe
5 environmental chamber
6 drive shaft
7 electro-dynamic vibration generator
8 force sensor
9 displacement sensor
10 driver input
11 instrument controls for force, displacement and driver units
12 computer
13 temperature probe

NOTE The drive shaft is rigidly attached to the sample clamp and vibration generator so motion is that of a shear beam.

Figure 1 — Schematic diagram of test apparatus

4.3 Displacement transducer

To eliminate inertial effects, a non-contacting sensor (typically an eddy current type or an optical encoder that is appropriately calibrated) with the following specifications shall be used to measure the specimen complex displacement, magnitude and phase:

— frequency range: 0,3 Hz to 30 Hz;

— uncertainty: $<$ 0,5 %.

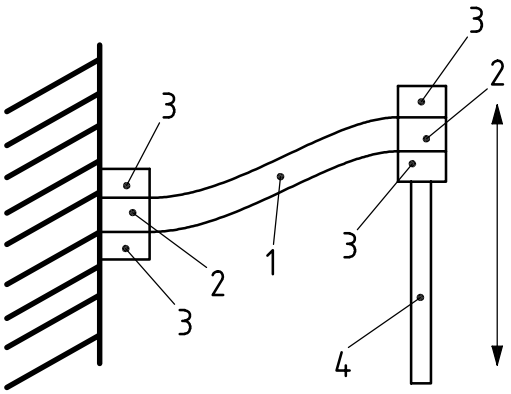
4.4 Clamping system

One end of the specimen is clamped rigidly to a frame using the attached end block. (See 5.1.) The driven end block is clamped into a fixture actuated by an electro-dynamic vibration generator via a rigid drive shaft.

The rigidity of the drive shaft and clamping fixture shall be tens to hundreds times larger that the bending stiffness of the specimen so that all of the measured displacement may be attributed to sample deformation.

This clamping system assures that the sample motion is confined to a cantilever shear beam mode with fixed-fixed ends. Figure 2 shows the required mode of deformation.

While in the past it was common not to use end blocks, their use has been found necessary in order to obtain reproducible and reliable results[1].



Key

1 beam specimen
2 specimen end blocks
3 specimen clamps
4 drive shaft

Figure 2 — Schematic diagram of sample deformation

4.5 Environmental chamber

An environmental chamber is required to cool the test sample to a temperature below room temperature. This temperature shall be maintained until the sample has reached equilibrium, then the temperature of the sample shall be increased in increments of typically 5 °C. The chamber should be capable of operating over the temperature range from − 60 °C to 70 °C and be controllable within 0,5 °C. The temperature sensor shall be appropriately calibrated.

NOTE 1 The required temperature range is appropriate for a visco-elastic material having a glass transition temperature greater than − 45 °C. Materials with lower glass transition temperatures will require a lower starting temperature point.

NOTE 2 Some materials are sensitive to humidity and it may be desirable to control or at least record the relative humidity in the chamber.

4.6 Computer

The use of a computer is advantageous to automate the calibration, data acquisition and processing.

5 Operating procedure

5.1 Sample preparation and mounting

5.1.1 General

Test specimens are typically cut from a sheet moulded or cast to the desired thickness using a small band saw or razor. It has been found that machining specimens from a thicker sample often affects the properties of the material. Specimens shall be uniform along each axis, and the ends shall be square to promote adhesion to the

end blocks. The dimensions of the specimen depend on the specific instrument and specimen stiffness. A typical specimen is 15 mm × 10 mm × 3 mm.

Three properties of the specimen, which are required in the analyses, shall be measured before bonding the specimen to the mounting blocks. In accordance with ISO 23529, determine the length, width and thickness, in metres, to four significant digits. The dimensions shall be measured at three locations along each axis then averaged.

5.1.2 Specimen end blocks

Steel or aluminium end blocks are attached to the ends of the specimen for clamping purposes. The actual dimensions of the end block vary with the clamping fixture configuration, but typical dimensions are 20 mm × 15 mm × 5 mm for the specimen in 5.1.1.

5.1.3 Specimen preparation

The specimen is bonded to the end blocks using a rigid adhesive. The elastic modulus of the adhesive shall be greater than that of the specimen and shall be stable over the experimental temperature range. Epoxy, urethane and cyanoacrylate adhesives have all been used successfully. Prior to bonding, the end blocks should be cleaned with denatured alcohol or other degreaser to promote adhesion. After the adhesive has cured, excess adhesive shall be carefully removed, taking care to avoid cutting the specimen or damaging the end block bond.

5.1.4 Specimen mounting

The specimen shall be mounted in the clamping fixture as shown in Figure 1. There are no set guidelines for the clamping fixture except that it should be rigid enough to insure the desired deformation. The specimen shall be placed in the fixture so that the clamps touch only the end blocks. The clamps shall be tightened sufficiently to eliminate slippage.

5.2 Conditioning

5.2.1 Storage

The time delay between moulding or vulcanization, testing and preconditioning of samples shall be in accordance with ISO 23529.

5.2.2 Temperature

Test pieces shall be thermally conditioned before each sequence of tests. At each test temperature, it is essential that the test piece be conditioned for sufficient time to reach equilibrium, but conditioning shall be no longer than necessary, particularly at higher temperatures, to avoid ageing effects. The conditioning time will depend on the test piece dimensions and the temperature. Guidance is given in ISO 23529.

5.2.3 Mechanical conditioning

Mechanical conditioning is generally omitted since only a single, very small strain is used as in free vibration applications. For large strains, the dynamic visco-elastic properties of many resilient materials are very dependent on the strain magnitude and temperature history. For such materials, it is recommended that the test pieces be preconditioned to obtain consistent and reproducible results. The test pieces shall be mechanically conditioned before testing to remove irreversible “structure”. The conditioning shall consist of at least six cycles at the maximum strain and temperature to be used in the series of tests. A minimum of 12 h rest period is required between mechanical conditioning and testing to allow reversible “structure” to equilibrate.

5.2.4 Humidity conditioning

Humidity is known to affect the physical properties of many resilient materials, especially urethanes. To ensure that measurements are made under reproducible conditions, samples shall be stored in a controlled-humidity environment for one week before testing. The controlled humidity is achieved by keeping the sample in a closed container that maintains a relative humidity of 50 % to 55 %. The temperature in the container shall be controlled between 20 °C and 25 °C during the conditioning period. Guidance is given in ISO 483.

5.3 Cantilever shear beam analysis

The basic principle of operation of the single cantilever shear beam apparatus is to determine the force needed to induce a measurable displacement of the specimen, as shown in Figure 2. As the magnitude of the displacement depends on the modulus of the specimen, this value may be calculated by relating force to displacement using the following equation[2]:

$$F\sin(\omega \cdot t) = M\,\frac{\mathrm{d}^2x}{\mathrm{d}t^2} + \left(\gamma + \frac{S''}{\omega} + \frac{kE''}{\omega}\right)\frac{\mathrm{d}x}{\mathrm{d}t} + (S' + kE')x \tag{1}$$

where

F is the peak force applied to the specimen (N);

$x(t)$ is the axial displacement of the drive shaft (m);

$\omega = 2\pi f$ is the angular frequency (rad/s);

M is the vibrating system mass (kg);

E' and E'' are the real and imaginary components of the Young's modulus of the specimen (Pa);

S' and S'' are the real and imaginary components of the system stiffness determined by calibration (Pa);

γ is the viscous damping coefficient, largely air, of the system (determined by calibration);

$k = w(t/l)^3/\left[1 + 2(1+\sigma)(t/l)^2\right]$ is a sample geometry factor (m)

where

w is the specimen width (m);

t is the specimen thickness (m);

l is the specimen length (m);

σ is Poisson's ratio for the specimen (typically assumed to be 0,45 to 0,49 for elastomers).

Defining the complex Young's modulus as

$$E^* = E' + iE'' = E'(1 + i\eta) \tag{2}$$

the solution of Equation (1) yields the elastic and loss moduli, E' and E'' respectively, as

$$kE' = K\cos\beta + M\omega^2 - S' \tag{3}$$

$$kE'' = K\sin\beta - S'' - \omega\gamma \tag{4}$$

where

β is the phase lag between stress and strain, including both material and test apparatus components;

$K = F/x$ is the effective stiffness of the specimen evaluated at peak values;

$\eta = E''/E'$ is the loss factor of the specimen.

NOTE 1 The loss factor, η, is sometimes referred to as the "tan δ " of the material.

NOTE 2 If the ratio t/l is small, the error in k due to the value of Poisson's ratio is small.

5.4 Calibration and measurement

The first step in the measurement procedure shall be to determine the complex stiffness of the vibration generator suspension and the viscous damping coefficient by making measurements with no specimen in place, i.e. $E' = E'' = 0$. Measurements shall be made both with and without an end block (which serves as an added mass) and at low and high frequencies, typically 1 Hz and 30 Hz. With the four measurements, the constants for the real and imaginary components of the system stiffness and the viscous damping coefficient shall be determined using Equation (1). The measurements shall be repeated ten times at each condition then averaged. The average values shall constitute the instrument calibration. A new calibration of the system's complex stiffness and viscous damping coefficient shall be made on a daily basis.

In each case, determine, using a balance, the vibrating system mass, in kilograms, to four significant digits.

Once the instrument has been calibrated, measurements shall be made with the specimen mounted as specified in 5.1. Force is applied to the specimen to attain a specific strain or displacement, typically 64 μm. This is repeated at a number of discrete frequencies, typically 0,3 Hz, 1 Hz, 3 Hz, 10 Hz and 30 Hz, and temperatures selected for the evaluation. Equation (1), with the value of k for the specimen geometry factor, shall be used to determine the complex Young's modulus at each frequency and temperature.

A data subset shall have a number of frequencies typically covering two decades at a single temperature. A complete determination of the dynamic mechanical properties of a resilient material shall consist of a number of subsets covering temperatures from the rubbery to glassy region.

NOTE The stiffness and frequency range specifications for these instruments vary depending on the specimen dimensions and the desired elastic modulus range. However, the following are provided for reference: Young's modulus range: 0,1 MPa to 3 GPa; stiffness range: 90 N/m to 15 MN/m; frequency range: 0,3 Hz to 30 Hz.

5.5 Number of test pieces

In order to obtain an indication of the variability of the material, it is recommended that a minimum of three representative samples be tested.

5.6 Temperature cycle

Measurements are made typically over the range from − 60 °C to 70 °C, using the following thermal cycle:

— cool the test specimen, mounted in the test apparatus, to − 60 °C;

— after reaching equilibrium, hold the specimen at this temperature to within ± 0,1 °C for at least 15 min before making any measurements;

— after each set of measurements, increase the temperature by no more than 5 °C;

— allow a minimum of 6 min to elapse after the air temperature has reached the new equilibrium temperature to within ± 0,1 °C before making the next measurement.

6 Analysis of results

6.1 Time-temperature superposition

Produce a reduced frequency plot of the modulus and loss factor data in the following manner.

a) Make graphical plots of the real part of the modulus as a function of the base 10 logarithmic frequency for each temperature.

b) Select as the reference temperature, T_0, the temperature for which the real part of the modulus has the greatest frequency slope.

c) Keeping the data at T_0 fixed, shift the real part of the modulus data for the other temperatures, in sequence, along the logarithmic frequency axis until each plot partially overlaps the previous data. Obtain the best fit of the overlap by minimizing the sum of the squares of the differences between sets of data at different temperatures. The magnitude of the shift required at each temperature is known as the shift factor a_T.

NOTE 1 The real part of the modulus is chosen to be shifted rather than the loss factor because the modulus is measured more accurately and has less scatter than the loss factor.

d) Shift the loss factor data using the same shift factor that was determined for the real part of the modulus.

NOTE 2 A material for which the above time-temperature superposition is applicable is called thermorheologically simple. A material which fails to superimpose, due to multiple transitions or crystallinity, for example, is thermorheologically complex.

e) The resulting plots of the real part of the modulus and loss factor as a function of shifted logarithmic frequency at reference temperature T_0 are known as master curves and span a wider range of frequency than measured.

NOTE 3 For a typical visco-elastic material, the shifted frequency range is from about 10^{-3} Hz to 10^{9} Hz.

f) Plot the Napierian logarithm of the shift factor, ln a_T, as a function of temperature. Fit these data to the Williams, Landel and Ferry (WLF) equation[3]:

$$\ln a_T = \frac{-c_{10}(T - T_0)}{(c_{20} + T - T_0)} \tag{5}$$

where c_{10} and c_{20} are constants for a given polymer, and subscript zero refers to the reference temperature T_0 at which the equation is evaluated.

g) The master curves at reference temperature T_0 are shifted to some other reference temperature, T_{ref}, as follows. Determine the logarithmic change in frequency corresponding to the temperature change from T_0 to T_{ref} by evaluating Equation (5) at the temperature T_{ref}. Subtract this logarithmic change in frequency from the values of the logarithmic frequencies corresponding to each of the data points obtained at T_0. The plot using the new frequencies is the master curve for T_{ref}.

NOTE 4 The lower limit in selecting a reference temperature is about equal to the glass transition temperature (T_g), while the upper limit is about T_g plus 100 °C. This upper limit is different for different polymers. The limits exist because the WLF equation only applies in the glass transition region.

6.2 Data presentation

Data obtained by the methods of this part of ISO 18437 shall be presented in the form of three graphs:

a) the base 10 logarithmic loss factor versus the base 10 logarithmic real part of the modulus;

b) shift factor versus temperature;

c) master curves of the base 10 logarithmic real part of the modulus and the base 10 logarithmic loss factor versus the base 10 logarithmic frequency at a specified reference temperature. Room temperature may be used for the reference temperature.

NOTE The plot of the logarithmic loss factor versus the logarithmic real part of the modulus includes all data without regard to temperature or frequency. This plot gives an indication of the consistency of the data. Points which do not lie along the curve are suspect and may be ignored. From its inverted “U” shape, this plot is sometimes referred to as a wicket plot.

In order to promote uniformity and ease in interpreting the data at temperatures other than the reference temperature, it is recommended that the master curves of the real and imaginary parts of the modulus and the loss factor be presented as a nomogram using the procedure given in ISO 10112.

6.3 Test report

The test report shall include the following information:

a) a reference to this part of ISO 18437;

b) all details necessary for complete identification of the material tested, including type, source, manufacturer's code number, form and previous history when these are known;

c) if applicable, the direction of any non-uniform feature of the test sample;

d) the date of the test;

e) the shape and dimensions of the test sample;

f) the method of preparation of the test samples;

g) details of the conditioning of the test samples;

h) the number of specimens tested;

i) details of the test atmosphere including humidity;

j) a description of the apparatus used for the test;

k) the temperature sequence used in the test, including the initial and final temperature as well as the rate of linear change in temperature or the size and duration of the temperature steps;

l) a table of the test results including the real and imaginary parts of the modulus and the loss factor versus frequency at each test temperature;

m) the modulus and loss factor versus frequency and temperature plots prepared as specified in 6.2.

Annex A
(informative)

Linearity of resilient materials

In principle the dynamic properties of a vibro-acoustic isolator are dependent on static preload, vibration magnitude, frequency and temperature.

The assumption of linearity implies that the principle of superposition holds and that the dynamic stiffness at a given frequency is independent of magnitude. For many isolators, this assumption is approximately satisfied when under the appropriate static preload, the dynamic deformation magnitudes are small compared with the static deformation. However, it should be noted that this depends on the materials of which the isolators are composed and a simple check should be carried out by comparing the dynamic stiffness characteristics for a range of input levels. If these are nominally invariant, then linearity may be assumed to hold.

For butyl rubber (IIR), Reference [4] presents data for the in-phase component and the phase angle of the dynamic shear modulus as a function of strain magnitude and of the percentage of carbon black. For strain magnitudes smaller than about 1,0 mm/m, the in-phase component and phase angle are hardly dependent on the vibration magnitude. However, a significant decrease of dynamic stiffness is seen when strain magnitudes exceed about 2,0 mm/m, especially for rubber with a high percentage of carbon black.

Therefore, it is important to consider strain magnitudes that occur in practice, and to check whether the test conditions are appropriate for the testing of rubber isolators. For strain magnitudes smaller than about 1,0 mm/m, the assumption of linearity (implying, for example, magnitude-independent stiffness and reciprocity) seems justified.

Hydraulic mounts are increasingly used, especially for automotive applications. This type of isolator may also show a very nonlinear behaviour, i.e. stiffness strongly dependent on vibration magnitude. Because of their two-fold purpose (i.e. damping of low-frequency engine vibration caused by road excitation and isolation of engine-generated structure-borne sound at higher frequencies), appropriate test magnitudes have to be applied for the whole frequency range of interest[5]-[8].

It is sometimes known *a priori* that linearity does not hold. In such cases it may still be advantageous to apply many of the procedures described in ISO 10846-1. Often this will imply that, in addition, special test requirements will be formulated with respect to preloads, signal magnitudes and measuring quantities.

Annex B
(informative)

Time-temperature superposition

It is presumed that a set of valid complex modulus data has been obtained in accordance with good practice. To check the consistency and scatter of the data, plot all data, regardless of frequency or temperature, on a plot of log (loss factor) versus log (modulus). This plot is commonly referred to as a wicket plot. If the data represent a thermorheologically simple material and if the data have no scatter, the data will plot as a single, smooth curve. As unshifted data are plotted in the wicket plot, no part of any scatter in this plot can be attributed to the shifting procedure.

While not required or used in the graphical presentation of data, the wicket plot is useful as a qualitative indication of the scatter of the experimental data. The width of the band of data, as well as the departure of individual points from the centre of the band, is indicative of scatter. Nothing is revealed about the accuracy of the temperature and frequency measurements or about any systematic error. The significance of time-temperature superposition is demonstrated through the concept of reduced frequency.

In general, the complex Young's modulus of a visco-elastic material is a function of frequency and temperature:

$$E^* = E^*(f, T) \tag{B.1}$$

In a thermorheologically simple material, these variables appear only as the product of frequency and a function of temperature known as the relaxation time:

$$E^* = E^*\left[f\tau(T)\right] \tag{B.2}$$

Hence a change in frequency is equivalent to a change in temperature. Consequently, the shift factor can be expressed as the ratio of the relaxation time at temperature T to the relaxation time at a reference temperature T_0:

$$a_T(T) = \tau(T)/\tau(T_0) \tag{B.3}$$

The complex modulus can be written as:

$$E^* = E^*\left[f a_T(T)\tau(T_0)\right] \tag{B.4}$$

and the reduced frequency is defined as:

$$f_\mathrm{R} = f a_T(T) \tag{B.5}$$

The complex modulus can be expressed in two equivalent ways:

$$E^* = E^*\left[f a_T(T)\tau(T_0)\right] = E^*\left[f_\mathrm{R}\tau(T_0)\right] \tag{B.6}$$

so that a modulus value measured at frequency f and temperature T is equivalent to a value at reduced frequency f_R and temperature T_0. The reduced frequency can be much greater than the measured frequency (by a factor a_T) since measurements made as a function of temperature are equivalent to measurements made over a wider frequency range than measured.

The shift factor has been presented here in a formal mathematical manner. The significance of the shift factor function, and the origin of its name, can be illustrated graphically. Consider a log-log plot of experimental measurements of E' versus frequency plotted as a series of isotherms. Pick one isotherm temperature as the reference temperature. The next highest isotherm can be shifted along the log frequency axis until it partially overlaps the reference isotherm. This process is continued with all the isotherms, in sequence, both higher and

lower than the reference. The result is a plot of log E' over a wide range of log reduced frequency values. This plot is known as a master plot. The amount of shift required to produce overlap can be plotted as a function of temperature. Because this function was generated by shifting data, it is known as the shift factor function. A plot of the shift factor can then be compared to the Williams-Landel-Ferry equation[3], [8].

Users of this part of ISO 18437 who are unfamiliar with extending the frequency range of their measurements by time-temperature shifting are referred to ANSI S2.24[9] for further guidance. In ANSI S2.24, the theory, sample data, the method of shifting, the resulting shift factor, analytical representation, and the graphical presentation of those data in the form of a frequency-temperature nomogram are presented for a standard material.

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