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

Part 4:
Dynamic stiffness method

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

Partie 4: Méthode de la raideur dynamique

Reference number
ISO 18437-4:2008(E)

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Foreword

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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 18437-4 was prepared by Technical Committee ISO/TC 108, *Mechanical vibration, shock and condition monitoring*.

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: Dynamic stiffness method*

The following parts are under preparation:

— *Part 1: Principles and guidelines*

— *Part 5: Poisson's ratio based on finite element analysis*

Introduction

Visco-elastic materials are used extensively to reduce vibration magnitudes, of the order of hertz to kilohertz, in structural systems through dissipation of energy (damping) or isolation of components, and in acoustical applications that require modification of the reflection, transmission, or absorption of energy. The design, modelling and characterization of such systems often require specific dynamic mechanical properties (the Young, shear, and bulk moduli and their corresponding loss factors) in order to function in an optimum manner. Energy dissipation is due to interactions on the molecular scale and can be 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 amplitude. 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, in principle, of the operation of the direct dynamic stiffness method, the measurement equipment used in performing the measurements, and the analysis of the resultant data. A further aim 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 amplitudes, although the static stiffness may be non-linear.

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

Part 4: **Dynamic stiffness method**

1 Scope

This part of ISO 18437 specifies a direct method for measuring the complex dynamic moduli of elasticity (the Young, shear and bulk moduli, and their respective loss factors corresponding to the tensile, shear and all compressive strains) for polymeric (rubbery and viscous polymers, as well as rigid plastics) materials over a wide frequency and temperature range. Measurements are performed by the dynamic stiffness method, which uses electric signals from sensors attached to a test piece. These signals are proportional to the dynamic forces acting on the test piece and the strains in the test piece due to the effect of these forces.

The measurement frequency range is determined by the size of test piece, the accuracy required on the dynamic modulus measurements, the relationship between the stiffness of the oscillation generator and the stiffness of the test piece, and by the resonance characteristics of the test fixture used.

The method presented in this part of ISO 18437 allows measurement under any static pre-load allowed for the test piece (including the test piece having the non-linear characteristics under different static loads), but under small dynamic (acoustic) strains, *i.e.*, in limits where the linear properties of the test piece are not distorted. Depending on the pre-load conditions, the relation between the moduli is unique.

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, *Plastics — Vocabulary*

ISO 483, *Plastics — Small enclosures for conditioning and testing using aqueous solutions to maintain the humidity at a constant value*

ISO 2041, *Mechanical vibration, shock and condition monitoring — Vocabulary*

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

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

ISO 6721-4, *Plastics — Determination of dynamic mechanical properties — Part 4: Tensile vibration — Non-resonance method*

ISO 6721-6, *Plastics — Determination of dynamic mechanical properties — Part 6: Shear vibration — Non-resonance method*

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

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

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

NOTE ISO 10846-1 is concerned with the global measurement of dynamic input and transfer stiffness and mechanical resistance of resilient fixtures. This part of ISO 18437 is concerned with the characterization of the dynamic Young modulus, shear modulus, bulk modulus, and corresponding loss factors of the visco-elastic materials that are used in the fixtures.

3 Terms and definitions

For the purposes of this part of ISO 18437, the terms and definitions given in ISO 472, ISO 483, ISO 2041, ISO 4664-1, ISO 6721-1, ISO 6721-4, ISO 6721-6, ISO 10112, ISO 10846-1, ISO 23529, and the following apply.

3.1
dynamic mechanical properties
⟨visco-elastic materials⟩ fundamental elastic properties, *i.e.*, elastic modulus, shear modulus, bulk modulus and loss factor

3.2
damped structure
structure containing elements made from damping materials

3.3
Young modulus
modulus of elasticity
E
ratio of the normal stress to linear strain

NOTE 1 Adapted from ISO 80000-4-18.1:2006[9].

NOTE 2 The Young modulus is expressed in pascals.

NOTE 3 The complex Young modulus, E^*, for a visco-elastic material is represented by $E^* = E' + \mathrm{i}E''$, where E' is the real (elastic) component of the Young modulus and E'' is the imaginary (loss modulus) component of the Young modulus. The real component represents elastically stored mechanical energy, while the imaginary component is a measure of mechanical energy loss.

3.4
shear modulus
modulus of rigidity
Coulomb modulus
G
ratio of the shear stress to the shear strain

NOTE 1 Adapted from ISO 80000-4-18.2:2006[9].

NOTE 2 The shear modulus is expressed in pascals.

NOTE 3 The complex shear modulus, G^*, for a visco-elastic material is represented by $G^* = G' + \mathrm{i}G''$, where G' is the real (elastic) component of the shear modulus and G'' is the imaginary (loss modulus) component of the shear modulus.

3.5
bulk modulus
modulus of compression
K
the negative ratio of pressure to volume strain

NOTE 1 Adapted from ISO 80000-4-18.3:2006[9].

NOTE 2 The bulk modulus is expressed in pascals.

NOTE 3 The complex bulk modulus is represented by $K^* = K' + iK''$, where K' is the real (elastic) component of the bulk modulus and K'' is the imaginary (loss modulus) component of the bulk modulus.

3.6
loss factor
ratio of the imaginary component to the real component of a complex modulus

NOTE When a material shows a phase difference, δ, between dynamic stress and strain in harmonic deformations, the loss factor is equal to $\tan\delta$.

3.7
magnitude of complex modulus
absolute value of the complex modulus

NOTE The magnitude of the complex moduli are defined as:

a) magnitude of the Young modulus: $|E| = \sqrt{[(E')^2 + (E'')^2]}$;

b) magnitude of shear modulus: $|G| = \sqrt{[(G')^2 + (G'')^2]}$;

c) magnitude of bulk modulus: $|K| = \sqrt{[(K')^2 + (K'')^2]}$.

These magnitudes are expressed in pascals.

3.8
frequency-temperature superposition
principle by which, for visco-elastic materials, frequency and temperature are equivalent to the extent that data at one temperature can be superimposed upon data taken at different temperature merely by shifting the data curves along the frequency axis

3.9
shift factor
measure of the amount of shift along the logarithmic axis of frequency for one set of data at one temperature to superimpose upon another set of data at another temperature

3.10
glass transition temperature
T_g
⟨visco-elastic materials⟩ temperature at which a material changes state reversibly from glassy to rubbery

NOTE 1 The glass transition temperature is expressed in degrees Celsius.

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 temperature higher than T_g and varies with the measurement frequency, hence it is not an intrinsic material property.

3.11
linearity
⟨visco-elastic materials⟩ property of dynamic behaviour of a resilient material if it satisfies the principle of superposition

NOTE 1 The principle of superposition can be 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 $\alpha y_1(t) + \beta y_2(t)$, where α and β are arbitrary constants. This must hold for all values of α, β and $x_1(t)$, $x_2(t)$.

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

4 Principle

The dynamic stiffness method is a technique for determining the frequency characteristics of the complex dynamic modulus of elasticity of resilient materials using small test pieces mounted in an appropriate test fixture.

Before performing the measurement, test pieces of the material are manufactured and placed in a test fixture where they are subjected to a strain with the help of a displacement actuator. The force transducer electric output is proportional to the force acting on the test piece; the displacement actuator electric input signal is proportional to the strain in the test piece. The test piece shall have dimensions such that its impedance is completely elastic in character over the total frequency range of interest. Hence the inertial component of this impedance shall be negligible in comparison with the elastic component. To meet this requirement, the test piece sizes shall be such that the first eigenfrequency should be three to five times larger than the upper frequency limit of measurement.

In the dynamic stiffness method, when using special fixtures, it is possible to apply the three different types of strain: the Young (tensile or compressive), shear, and bulk to the test piece and thus measure the three corresponding moduli of elasticity and their corresponding loss factors (when the displacement actuator generates deformation only along the test piece axis). The user can choose a test piece shape and fixture for applying an appropriate type of strain in each specific case.

When performing the measurement using the specific conditions detailed above, the general expression for determination of the complex elastic modulus, $E^*,G^*,K^*(f)$, has the form

$$E^*,G^*,K^*(f) = \alpha_{E,G,K}[F(f)/s(f)]. \tag{1}$$

where

$\alpha_{E,G,K}$ is the ratio of the measured modulus of the tested material to stiffness of the test piece under the appropriate strain (longitudinal, shear or bulk);

NOTE Methods of calculating $\alpha_{E,G,K}$ are shown in Clause 6.

$F(f)/s(f)$ is the complex ratio of the output force and the test piece displacement.

Hence, the real part of the modulus, E', G', $K'(f)$, is given by Equation (2):

$$E',G',K'(f) = \alpha_{E,G,K}\,\mathrm{Re}[F(f)/s(f)] \tag{2}$$

The imaginary part of the modulus, $E'',G'',K''(f)$, is given by Equation (3):

$$E'',G'',K''(f) = \alpha_{E,G,K}\,\mathrm{Im}[F(f)/s(f)] \tag{3}$$

The magnitude of the modulus, $\left|E,G,K(f)\right|$, is given by Equation (4):

$$\left|E,G,K(f)\right| = \alpha_{E,G,K} \left|F(f)/s(f)\right| \tag{4}$$

The loss factors, $\eta_{E,G,K}(f)$, are given by Equation (5):

$$\eta_{E,G,K}(f) = \mathrm{Im}[F(f)/s(f)]/\mathrm{Re}[F(f)/s(f)] \tag{5}$$

5 Equipment

5.1 Hardware

The following items are used for carrying out the measurements:

5.1.1 2-Channel fast Fourier transform (FFT) analyser, which provides a measurement of complex value frequency response function.

5.1.2 Input and output transducer, and **preamplifiers** as required.

5.1.3 Computer.

5.1.4 Test device and **test piece**, including force transducer and displacement actuator.

A temperature sensor (such as a thermocouple or thermostat) shall be placed in the test device when temperature dependence of moduli and loss factors is to be measured. The device for controlling the temperature of the test piece may be mounted inside the test device. The thermostat shall measure the actual temperature of the test piece over the range –60 °C up to +70 °C, at minimum increments of 5 °C.

5.2 Set-up

A typical measurement set-up and test device for measuring the visco-elastic characteristics, such as the dynamic moduli of elasticity and loss factors, of a polymeric material are shown in Figure 1 and Figure 2 respectively (Reference [1]). Depending on the test device and the material, the frequency range can be up to 10 kHz.

If the application of the visco-elastic material is for structure-borne noise or vibration suppression, it should be tested up to 500 Hz (see ISO 10846-1).

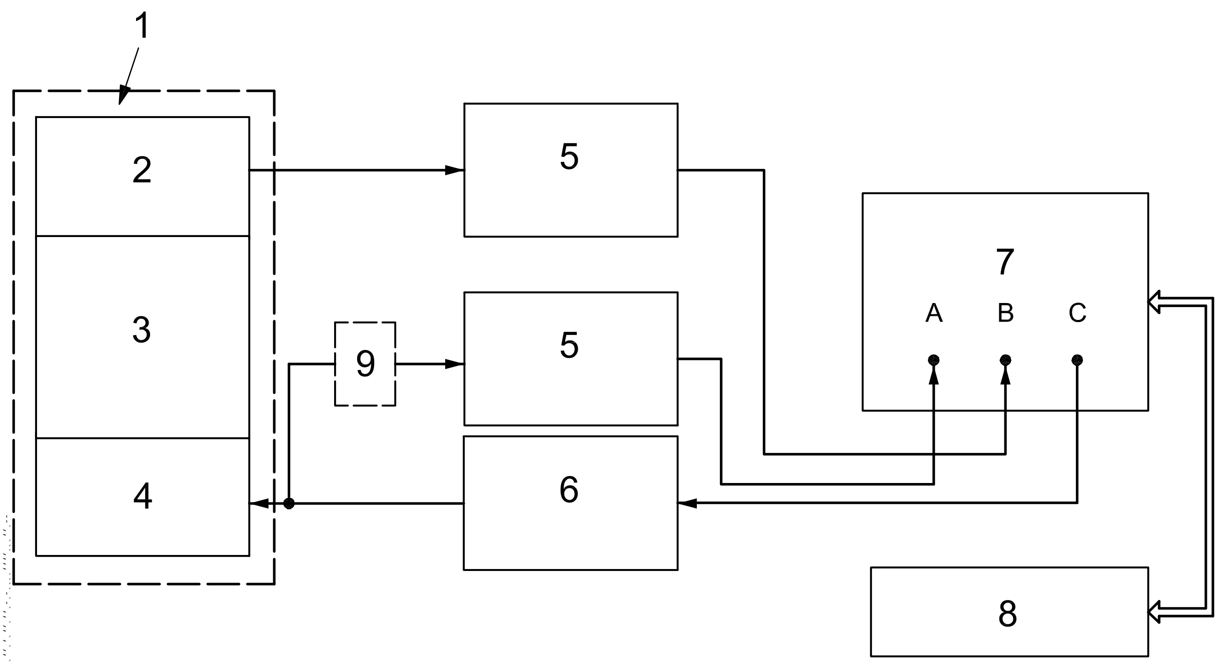
The test set-up comprises the following components:

- rigid restrictive construction;
- means of fixing or attaching test pieces to the test set-up;
- two electromechanical units, a displacement actuator and a force transducer — the former converts the electrical signal from the power amplifier into a surface displacement that is in contact with the test piece and deforms it, while the latter converts the force acting on the test piece into an electric signal (see Figure 2);
- annular washers for adjustment of the gap between the force transducer and the displacement actuator when carrying out test piece measurements under any permissible static pre-load;
- external fixture to generate a known static compression in the test piece when attached to the electromechanical units.

In the following, the numbers in parentheses refer to the labels in Figure 2. The test piece (3) rigidity shall be far less than the rigidity of the displacement actuator (1), force transducer (2), annular washer (4) and the rigid restrictive construction (6).

The test piece (3) is placed between operational surfaces of displacement actuator and force transducer.

When measuring the characteristics of the test piece under a pre-load, if required for testing, the distance between the support surfaces of the cylindrical shells shall be adjusted by the annular washer (4). These washers are parallel to the test piece surfaces. When the objective is measurement under zero static displacement, the height of the annular washer shall be 3 % to 5 % less than that of the test piece. This arrangement produces zero static pre-load.



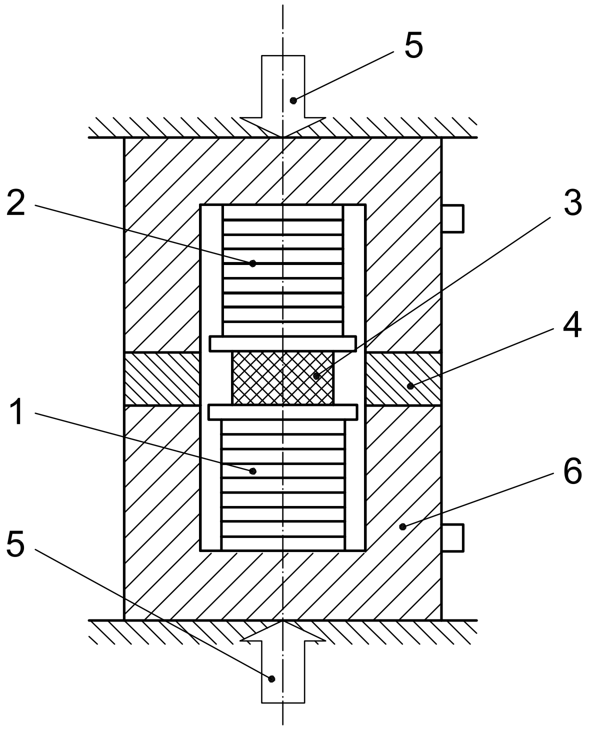
Key

1 test device
2 force transducer
3 test piece
4 displacement actuator
5 amplifier [a]
6 power amplifier
7 dual channel spectrum FFT analyser [b]
8 PC
9 voltage divider
A channel A — displacement from displacement actuator input
B channel B — force from force transducer output
C channel C — excitation signal from the FFT analyser

[a] The amplifier shall have the functions of amplification and attenuation of the signal. If the signal from the output of the power amplifier is too large for the amplifier, a voltage divider shall be added before the amplifier. The voltage divider shall not distort the signal's phase by more than 0,05°.

[b] Channel A is the displacement from displacement actuator input, channel B is the force from force transducer output and channel C is the excitation signal from the FFT analyser.

Figure 1 — Measurement set-up



Key

1 displacement actuator
2 force transducer
3 test piece and nearby temperature sensor
4 annular washer
5 static pressure-generating fixture
6 rigid restrictive construction

Figure 2 — Schematic diagram of the measurement device

When measuring the unknown values by using the test device shown in Figure 2, the real part of each modulus, $E',G',K'(f)$, is given by Equation (6):

$$E',G',K'(f) = \alpha_{E,G,K}\,\mathrm{Re}[\beta(f)\cdot H(f)] \tag{6}$$

where $\beta(f)$ is defined in Equation (10) and $H(f)$. in Equation (11).

The imaginary part of the modulus, $E'',G'',K''(f)$, is given by Equation (7):

$$E'',G'',K''(f) = \alpha_{E,G,K}\,\mathrm{Im}[\beta(f)\cdot H(f)] \tag{7}$$

The magnitude of the modulus, $\left|E,G,K\left(f\right)\right|$, is given by Equation (8):

$$\left|E,G,K\left(f\right)\right| = \alpha_{E,G,K}\left[\left|H\left(f\right)\right| / \left|K_s\right|\cdot\left|\lambda_F\right|\right] \tag{8}$$

The loss factors, $\eta_{E,G,K}(f)$, are given by Equation (9):

$$\eta_{E,G,K}(f) = \mathrm{Im}[\beta(f)\cdot H(f)]/\mathrm{Re}[\beta(f)\cdot H(f)] \tag{9}$$

The complex function, $\beta(f)$, describes the characteristics of the displacement actuator and the force transducer of the test device in the absence of the annular washer and test piece, and is determined using Equation (10):

$$\beta(f) = \frac{\cos\Delta\varphi(f) - \mathrm{i}\sin\Delta\varphi(f)}{|K_s| \cdot |\lambda_F|} \tag{10}$$

where

$|K_s|$ is the modulus of the transformer quotient, in metres per volt, of the displacement actuator;

$|\lambda_F|$ is the modulus of the force transducer sensitivity with respect to the applied force, in volts per newton;

$\Delta\varphi(f)$ is the phase angle, in degrees, between the signals at the output of the force transducer and the input of the displacement actuator if their measurement surface is in rigid mechanical contact.

These quotients are determined during the calibration of the force transducer and the displacement actuator.

The complex function, $H(f)$, when a test piece is placed into the device (see Figure 1), is given by Equation (11):

$$H(f) = U_F(f)/U_s(f) \tag{11}$$

where

$U_F(f)$ is the complex signal, in volts, at the output of the force transducer;

$U_s(f)$ is the complex valued input, in volts, of the displacement actuator.

Signal, $U_F(f)$, is applied through the amplifier to channel A of the two-channel analyser; input, $U_s(f)$, is applied to channel B of the two-channel analyser.

When using this type of test device, measurement errors do not exceed 2,5 % to 3,0 % in the frequency range 10 Hz to 10 kHz, if the measuring devices have the metrological characteristics shown in Table 1.

Table 1 — Characteristics of measuring equipment

Measurement tools and equipment	Specifications for measurement process	
	Frequency and voltage range	Accuracy
Dual channel FFT analyser, equipped with signal generator (random noise, sine wave)	5 Hz to 10 kHz 100 μV to 100 V (RMS)	Response ripple 2 % Channel phase $<$ 0,02°
Amplifiers	10 Hz to 10 kHz; electric noise level $\leqslant$ 5 μV	Response ripple 1 % Input/output phase difference $<$ 0,1°
Power amplifier	10 Hz to 10 kHz	Non-linear distortions $<$ 10 %

If the phase responses of measurement tools are different from those given in Table 1, such responses should be taken into account during the signal processing.

6 Recommended set-up for applying the different types of strain to the test piece and calculation of quotients, $\alpha_{E,G,K}$

6.1 Choosing test piece size

Test piece sizes should be chosen according to the conditions for ensuring:

- the error allowed when measuring the moduli of elasticity and loss factor for polymeric damping material;
- the required upper frequency limit, f_{max} = 10 kHz;
- the test piece shall not collapse under maximum test pre-load.

6.2 Rigid plastics

6.2.1 The Young modulus of rigid plastics

Cylindrical test pieces are recommended for measuring the Young moduli and the corresponding loss factors for rigid plastics (see Figure 3).

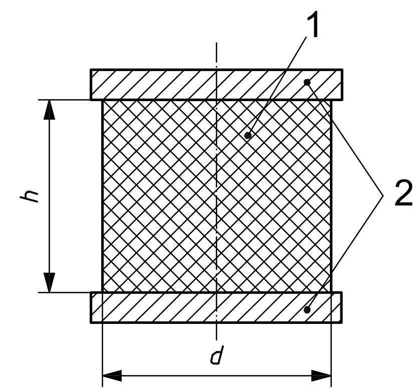
Under these conditions, the quotient, α_E, in Equation (1) is given by Equation (12):

$$\alpha_E = \frac{4h}{\pi d^2} \qquad (12)$$

where

h is the height of the cylindrical test piece;

d is the diameter of the cylindrical test piece.



Key

1 test piece
2 washers

d diameter
h height

Figure 3 — Test device for the Young modulus measurement

6.2.2 Shear modulus of rigid plastics

For the shear modulus and the corresponding loss factor measurement, parallelepipeds as shown in Figure 4 are required. The test piece sizes should be chosen so that their total shear stiffnesses are within the dynamic range of the test device.

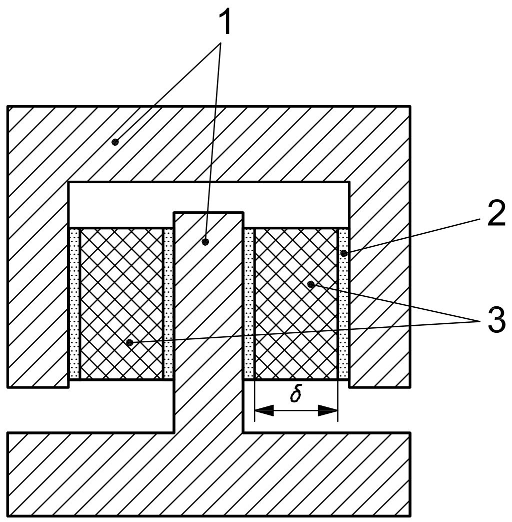
The quotient, α_G, of Equation (1) is given by Equation (13):

$$\alpha_G = \frac{\delta}{2lb} \quad (13)$$

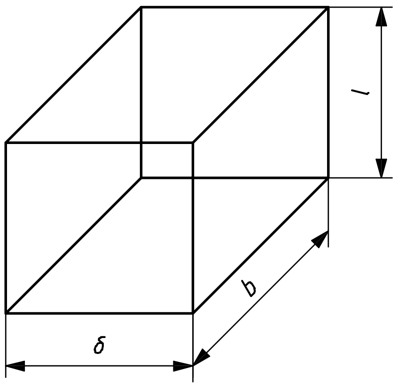
for a rectangular area of length, l, in the direction of the applied load, breadth, b, and thickness, δ; and for a square area by Equation (14):

$$\alpha_G = \frac{\delta}{2l^2} \quad (14)$$

The test piece length shall be at least 4 times larger than the thickness in order to make the correction for bending small (see ISO 6721-6 and Reference [4]).



a) test device



b) test piece

Key

1 covering straps

2 plate

3 test pieces

Figure 4 — Shear modulus measurement

6.3 Rubbery materials

6.3.1 The Young modulus of rubbery materials

For cylindrical test pieces, the quotient, α_E, in Equation (1) is given by Equation (15) (References [2], [3]):

$$\alpha_E = \frac{4h}{\pi d^2}\left[\frac{1}{1+(d^2/8h^2)}\right] \quad (15)$$

where

d is the diameter;

h is the height.

6.3.2 Shear modulus of rubbery materials

The measurement of shear modulus typically requires parallelepipedic test pieces. The sizes of the test pieces shall be chosen using the estimated modulus of elasticity so that the total stiffness of the test pieces is within the limits of the dynamic measurement range of the test device. The quotient, α_G, in Equation (1) for shear modulus measurement is given by Equation (16):

$$\alpha_G = \frac{\delta}{2lb} \tag{16}$$

for a rectangular cross-section test piece with length, l, breadth, b, and thickness, δ; for a square cross-section, the quotient is calculated from Equation (17):

$$\alpha_G = \frac{\delta}{2l^2} \tag{17}$$

The test piece length shall be at least 4 times larger than the thickness in order to make the correction for bending small (see ISO 6721-6 and Reference [4]).

6.4 Viscous materials

6.4.1 The Young modulus of viscous materials

For cylindrical test pieces, the quotient, α_E, in Equation (1) is given by Equation (18) (Reference [5], p. 99-100):

$$\alpha_E = \frac{4h}{\pi d^2}\left[\frac{1}{0{,}667 + (d^2/8h^2)}\right] \tag{18}$$

where

d is the diameter;

h is the height.

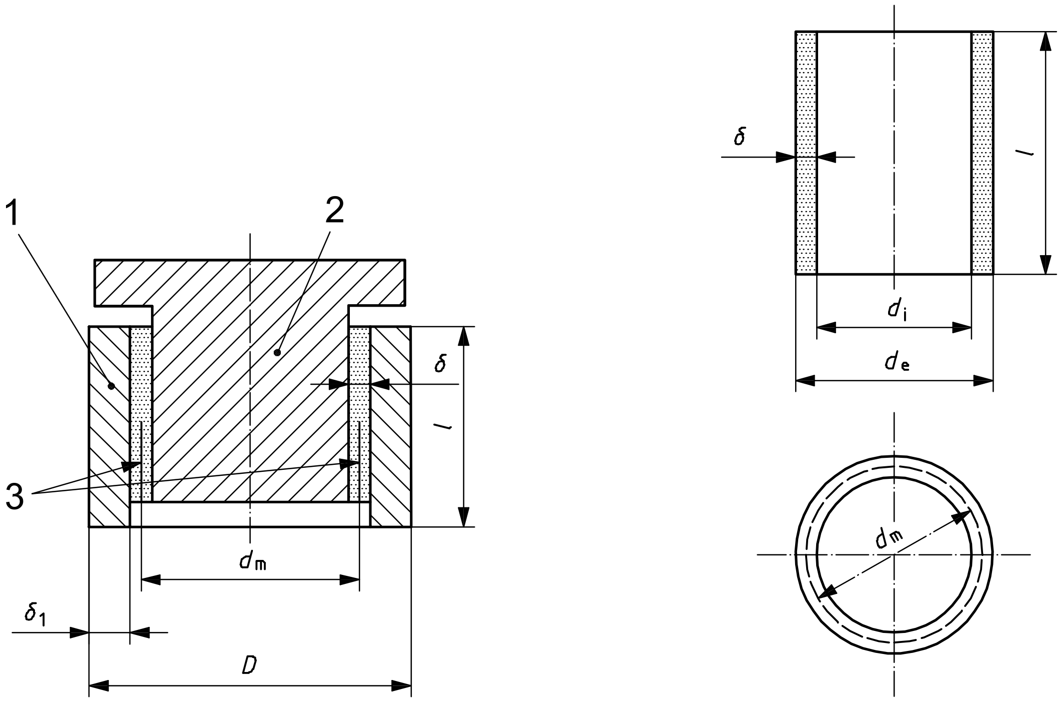
6.4.2 Shear modulus of viscous materials

The shear modulus and corresponding loss factor for the viscous material can be measured by a procedure similar to that for a rubbery material. The quotient, α_G, is determined using Equations (16) and (17). In this case, the complex moduli and corresponding loss factor are measured using the fixture shown in Figure 4.

If the arrangement shown in Figure 4 does not provide the required upper frequency limit, an alternative fixture shown in Figure 5 may be used. In this case, the test piece has the shape of cylindrical pipe.

The viscosity of some materials is such that it is possible to measure their elastic modulus using only the set-up shown in Figure 5, not the one shown in Figure 4.

The alternative fixture shown in Figure 5 is used when testing visco-elastic plastics. The test piece is made by pressing the material under test between the piston and the supporting cylinder.



a) test device b) test piece

Key

1 supporting cylinder
2 piston
3 test piece

δ pipe wall thickness

δ_1 supporting cylinder thickness

D outside diameter of supporting cylinder

d_m pipe mean diameter, $(d_e + d_i)/2$

d_e pipe external diameter

d_i pipe internal diameter

l test piece length

Figure 5 — Alternative shear modulus determination

Here the quotient, α_G, in Equation (1) is given by Equation (19):

$$\alpha_G = \frac{\delta}{\pi d_m l} \tag{19}$$

where

δ is the pipe wall thickness;

d_m is the pipe mean diameter, equal to $(d_e + d_i)/2$ (see Figure 5);

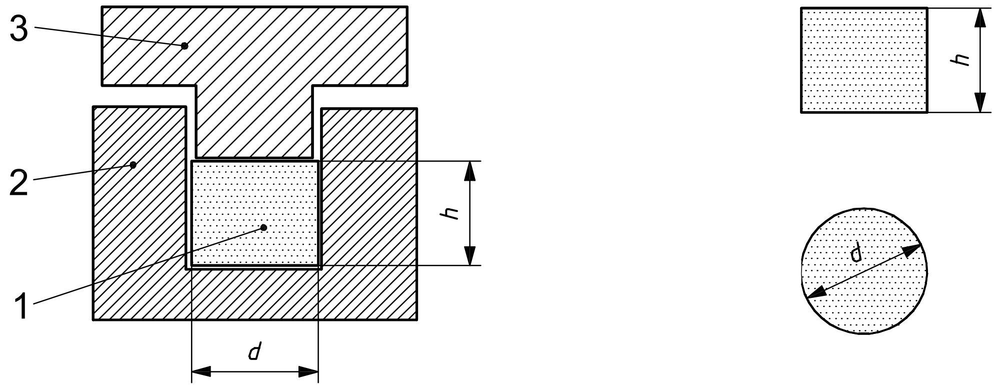
l is the length of the test piece.

6.5 Bulk modulus of all materials

To measure the bulk modulus of elasticity and corresponding loss factor of plastic, rubbery or viscous materials, solid cylindrical test pieces can be inserted into the fixture shown in Figure 6. Here the constant, α_K, in Equation (1) is given by Equation (20):

$$\alpha_K = \frac{4h}{\pi d^2} \quad (20)$$

NOTE At low pressures the measured values will be less than the bulk modulus until complete hydrostatic stress is achieved in the test piece.



a) test device b) test piece

Key

1 test piece
2 housing
3 piston

d diameter
h height

Figure 6 — Bulk modulus measurement

7 Test pieces

7.1 Choosing the shape and size of the test piece

The shape and size of damping material test pieces shall have:

a) stiffness values within the dynamic range of the test device;

b) maximum linear sizes such that spring-like behaviour is ensured within the frequency range of interest.

The test piece size shall be consistent with the modulus of the material and the test apparatus used. Generally, thick test pieces are appropriate for low modulus materials and thin test pieces for high modulus materials.

7.2 Instructions for manufacturing and preparing test pieces

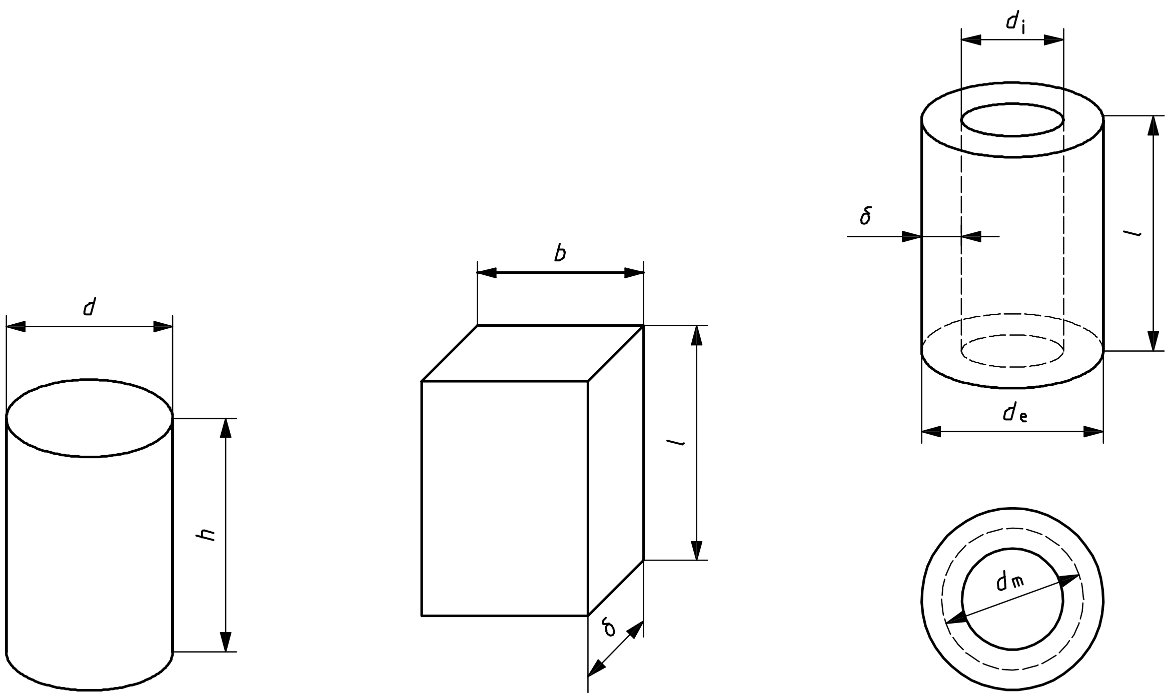
7.2.1 Introduction

The various test pieces for measuring the complex dynamic Young, shear and bulk moduli, and corresponding loss factors are shown in Figure 7.

The technology for manufacturing the test pieces from rubbery material is developed together with the material designer.

The test pieces of viscous polymeric materials are made using commercially manufactured products.

The technology for manufacturing the test pieces from viscous polymeric material is developed together with the material designer.



a) solid cylinder **b) rectangular parallelepiped** **c) hollow cylinder (pipe)**

Key

δ thickness

b breadth

d diameter

d_m pipe mean diameter, $(d_e + d_i)/2$

d_e pipe external diameter

d_i pipe internal diameter

h height

l length

Figure 7 — Test piece geometry for complex dynamic elastic moduli measurement

7.2.2 Test piece shape and preparation for the Young modulus determination

For measurement of the complex Young modulus and corresponding loss factors, a cylindrical test piece is required.

For measurement of the complex Young modulus of rigid plastics, it is recommended that Condition (21) be satisfied:

$$\frac{h}{d} \geqslant 1{,}0 \tag{21}$$

For measurement of the complex Young modulus of rubbery and viscous materials, to increase measurement accuracy (References [6], [7]), it is recommended that Condition (22) be satisfied:

$$\frac{h}{d} \geqslant 2{,}5 \tag{22}$$

After mechanical shaping, the face planes of cylindrical test pieces are polished to obtain a given height and to assure that the edges are parallel. Test piece sizes are measured to within ± 0,005 mm. The metallic plates that are glued to the test piece face planes before testing should be as thin as possible.

A rigid glue, which does not change the material structure, shall be applied (*e.g.*, cyanoacrylate). The glue layer shall be as thin as possible. The stiffness of the glue layers should be at least 10 times higher than expected actual stiffness of the test piece.

7.2.3 Test piece shape and preparation for shear modulus determination

Two test pieces, each of the same dimensions, in the form of rectangular parallelepipeds [see Figure 7b)] or cylindrical pipes [see Figure 7c)] are required for the measurement of shear modulus and corresponding loss factors (see Clause 4).

According to ISO 6721-6, unless $l \gg \delta$, there is a correction for thickness, so the test piece in both Figure 7b) and c) shall be thin.

The face planes of the parallelepiped test piece are polished smooth until the chosen sizes are obtained and the faces and edges are parallel. The measurement of test piece size is performed to within ± 0,005 mm.

The metallic plates that are glued to the test piece face planes before testing should be as thin as possible.

A rigid glue, which does not change the material structure, shall be applied. The glue layer shall be as thin as possible. The stiffness of the glue layers should be 10 times higher than expected actual stiffness of the test piece.

Test pieces shaped as a cylindrical piece of pipe are glued to the internal lateral surface of the supporting cylinder and to the external lateral surface of piston (see Figure 5). If the plasticity of the tested material is sufficient, one may use a forming method by filling the working space between the piston and the supporting cylinder of the test device.

For cylindrical pipe test pieces, the stiffness, C_G, values [see Figure 7c)] due to shear strain along the central cylindrical axis are given by Equation (23):

$$C_G = \frac{2G\pi d_\mathrm{m} l}{\delta} \tag{23}$$

where

δ is the pipe wall thickness, equal to $d_\mathrm{e} - d_\mathrm{i}$;

d_m is the pipe mean diameter, equal to $(d_e + d_i)/2$;

G is the shear modulus;

l is the length of the test piece.

7.2.4 Test piece shape and preparation for bulk modulus measurement

Solid cylindrical test pieces, of height, h, and diameter, d, are required for the measurement of the bulk modulus and the corresponding loss factors [see Figure 7a)].

Measurements of bulk modulus, K, shall be performed on test pieces with the minimum possible values of h. This condition facilitates the technology and improves test piece mounting (*e.g.*, by optimizing contact with elements of test device and eliminating air from the test zone), thus removing potential sources of error.

8 Conditioning

8.1 Storage

The time delay between molding or vulcanization and testing and pre-conditioning of the test pieces shall be in accordance with ISO 23529.

8.2 Temperature

Test pieces shall be thermally conditioned before a sequence of tests. At each test temperature, it is essential that the test piece is conditioned for sufficient time to reach equilibrium, but conditioning need 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.

8.3 Mechanical conditioning

The test pieces shall be mechanically conditioned before testing to remove irreversible “structure”. The conditioning should consist of at least six cycles at the maximum strain and temperature to be used in the series of tests. A minimum rest period of 12 h is required between mechanical conditioning and testing to allow reversible “structure” to equilibrate.

This mechanical conditioning can generally be omitted when very small strains are used.

8.4 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, test pieces shall be stored in a controlled humidity environment for one week before testing. The controlled humidity is achieved by keeping the test piece in a closed container that maintains a relative humidity of 50 % to 55 %.

8.5 Measurement conditioning

The measurements are conducted under normal conditions in a room at a temperature of 20 ± 5 °C containing air at a relative humidity 50 % to 55 %.

9 Main error sources

When measurements are performed by the dynamic stiffness method, possible causes of error are:

a) improper acoustical contact;

b) inaccuracy when manufacturing the test pieces;

c) incorrect choice of test piece sizes;

d) large phase shift between the device channels for measuring the values of forces and strains.

If acoustical contact between the test device and the test piece is insufficient (loose), measured stiffness values can be less than the actual value.

To ensure proper acoustic contact when measuring the test material, it is recommended that preliminary measurements be made to achieve the independence of measured stiffness from static strain. The static test piece displacement is determined as the difference between the test piece height and the total thickness of annular washers.

Errors connected with manufacturing the test piece can lead to the appearance of displacements, which are different from those measured. Therefore the measurement results cannot be attributed to the specified sort of displacement. To avoid these errors, it is necessary to achieve the required parallelism of contact surfaces with the test device, and also to assure the test piece's symmetry for eliminating test piece misalignments when the static compression operates.

Failure to comply with test piece size requirements (7.1) may result in errors, for example:

- if the test piece stiffness is comparable to that of the test device, the stiffness properties of the test device influence the measurement results;
- if the test piece stiffness is small, the force transducer produces a signal which is comparable to background noise of test device;
- if the condition for the stiffness characteristics of the test piece impedance is not fulfilled, test piece resonance influences measurement.

10 Measurement results and processing

10.1 Frequency-temperature superposition

In some cases, it may be desirable to determine the properties over a frequency range that is larger than that which is experimentally available. Frequency-temperature superposition is the process in which measurements at a number of temperatures are used to increase the frequency range.

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

1) make graphical plots of the real part of the modulus as a function of logarithms to the base 10 of frequency for each temperature;

2) select as the reference temperature, T_{ref}, the temperature for which the real part of the modulus has the greatest frequency slope;

3) keeping the data at T_{ref} 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 to obtain the best fit, calculated using the method of least squares — 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 shifted rather than the loss factor because the real part of the modulus is measured more accurately and has less scatter than the loss factor.

4) 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 frequency-temperature superposition is applicable is called thermorheologically simple. A material which fails to superimpose, due to multiple transitions or crystallinity for example, are thermorheologically complex.

5) the resulting plots of the real part of the modulus and loss factor as a function of shifted logarithmic frequency at T_{ref} are known as master curves and they span a wider range of frequency than measured;

NOTE 3 For a typical visco-elastic material, the shifted frequency range is from about 10^{-5} Hz to 10^{10} Hz.

6) plot the logarithm of the shift factor as a function of temperature, and fit these data to Equation (24) (see Reference [8], p. 287-298):

$$\lg a_T = \frac{-c_{10}(T - T_{ref})}{c_{20} + T - T_{ref}} \tag{24}$$

where

c_{10}, c_{20} are constants for a given polymer,

T_{ref} is the reference temperature at which the equation is evaluated;

7) the master curves at T_{ref} can be shifted to some other reference temperature, T'_{ref}, as follows. Determine the logarithmic change in frequency corresponding to the temperature change from T_{ref} to T'_{ref} by evaluating Equation (24) at a temperature, $T = T'_{ref}$. Subtract this logarithmic change in frequency from the values of the logarithmic frequencies corresponding to each of the data points obtained at T_{ref}. The plot using the new frequencies is the master curve for T'_{ref} .

NOTE 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 + 100) °C. This upper limit is different for different polymers. The limits exist because Equation (24) only applies in the glass transition region.

10.2 Data presentation

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

1) logarithm to the base 10 of loss factor versus logarithm to the base 10 of modulus magnitude using the procedure given in ISO 10112;

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

2) shift factor versus temperature;

3) master curves of logarithm to the base 10 of real part of the modulus and logarithm to the base 10 of loss factor versus logarithm to the base 10 of frequency at a specified T_{ref}. Room temperature may be used as T_{ref}.

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.

10.3 Test report

The test report shall include at least 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 piece;

d) the date of the test;

e) the shape and dimensions of the test piece;

f) the method of preparation of the test pieces;

g) details of the conditioning of the test pieces;

h) the number of test pieces tested;

i) details of the test atmosphere, including humidity and temperature;

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 loss factor versus frequency at each test temperature;

m) the real part of the modulus and loss factor versus frequency and temperature plots.

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