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Rubber, vulcanized or thermoplastic — Determination of dynamic properties —

Part 1: General guidance

Caoutchouc vulcanisé ou thermoplastique — Détermination des propriétés dynamiques —

Partie 1: Lignes directrices

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This third edition cancels and replaces the second edition (ISO 4664-1:2011), which has been technically revised.

The main changes are as follows:

— other types of deformation mode have been included in Table 2;

— descriptions of nonlinear behaviour have been added in 6.1.2;

— explanations regarding the forced resonant vibration type method have been added in 6.2;

— other shapes and dimensions of test pieces have been added in Table 4 (the former Table 3);

— test conditions (temperature, frequency, strain, etc.) have been expanded in Table 5 (the former Table 4);

— the derivation method for required viscoelastic parameters has been clarified in 9.5;

— test methods for free vibration and forced vibration resonant type have been detailed in Clauses 10 and 11.

A list of all parts in the ISO 4664 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rubber, vulcanized or thermoplastic — Determination of dynamic properties —

Part 1: General guidance

1 Scope

This document gives guidance on the determination of dynamic properties of vulcanized and thermoplastic rubbers. It includes both free- and forced-vibration methods carried out on both materials and products. It does not cover rebound resilience or cyclic tests in which the main objective is to fatigue the rubber.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1 Terms applying to any periodic deformation

3.1.1
hysteresis loop
closed curve representing successive stress-strain states of a material during a cyclic deformation

3.1.2
energy loss
energy per unit volume which is lost in each deformation cycle, i.e. the hysteresis loop area

Note 1 to entry: It is expressed in J/m^3.

3.1.3
power loss
energy loss (3.1.2) per unit time, per unit volume, which is transformed into heat through hysteresis, expressed as the product of energy loss and frequency

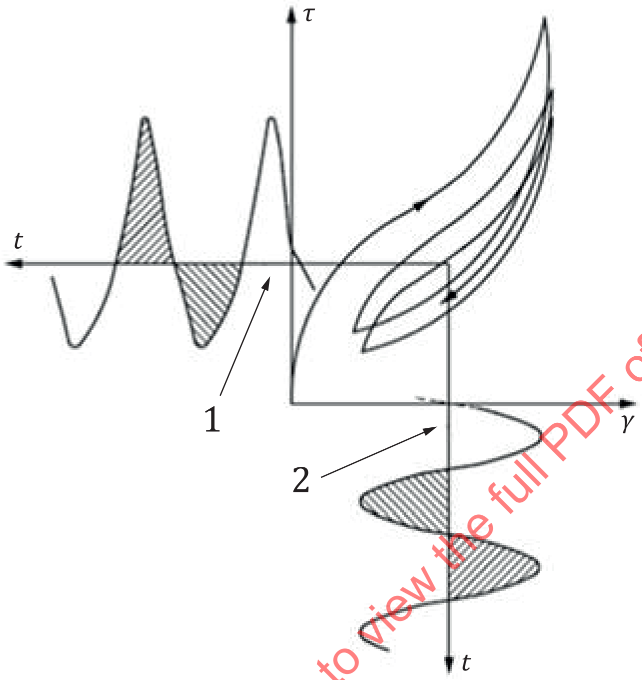
Note 1 to entry: It is expressed in W/m^3.

3.1.4
mean stress
average value of the stress during a single complete *hysteresis loop* (3.1.1)

Note 1 to entry: It is expressed in Pa.

Note 2 to entry: This is the static stress applied before starting dynamic motion.

Note 3 to entry: See Figure 1.



Key

1	mean stress	τ	stress
2	mean strain	γ	strain
		t	time

NOTE 1 Open initial loops are shown, as well as equilibrium mean strain and mean stress as time-averages of instantaneous strain and stress.

NOTE 2 A sinusoidal response to a sinusoidal motion implies hysteresis loops which are or can be considered to be elliptical.

NOTE 3 For large sinusoidal deformations, the hysteresis loop will deviate from an ellipse since, for rubber, the stress-strain relationship is nonlinear and the response is, therefore, not sinusoidal.

NOTE 4 The term "incremental" may be used to designate a dynamic response to sinusoidal deformation about various levels of mean stress or mean strain (for example, incremental spring constant, incremental elastic shear modulus).

Figure 1 — Heavily distorted hysteresis loop obtained under forced pulsating sinusoidal strain

3.1.5
mean strain
average value of the strain during a single complete *hysteresis loop* (3.1.1)

Note 1 to entry: This is the static strain applied before starting dynamic motion.

Note 2 to entry: See Figure 1.

3.1.6
maximum load amplitude
F_0
maximum applied load, measured from the average value of the load during a single sinusoidal wave

Note 1 to entry: It is expressed in N.

3.1.7
maximum stress amplitude
τ_0
ratio of the maximum applied force, measured from the mean force, to the cross-sectional area of the unstressed test piece (zero to peak on one side only)

Note 1 to entry: It is expressed in Pa.

3.1.8
maximum deflection amplitude
x_0
maximum value of the deflection, measured from the average value of the deflection during a single sinusoidal wave

Note 1 to entry: It is expressed in m.

3.1.9
maximum strain amplitude
γ_0
maximum value of the strain, measured from the *mean strain* (3.1.5) (zero to peak on one side only)

3.1.10
Payne effect
phenomenon in which the dynamic modulus decreases as the strain increases, in dynamic testing of a filled rubber compound

3.2 Terms applying to sinusoidal motion

3.2.1
spring constant
K
component of the applied load, which is in phase with the deflection, divided by the deflection

Note 1 to entry: It is expressed in N/m.

3.2.2
elastic shear modulus
storage shear modulus
G'
component of the applied shear stress, which is in phase with the shear strain, divided by the strain

$$G' = \left|G^*\right| \cos\delta$$

Note 1 to entry: It is expressed in Pa.

3.2.3
loss shear modulus
G''
component of the applied shear stress, which is in quadrature with the shear strain, divided by the strain

$$G'' = \left|G^*\right| \sin\delta$$

Note 1 to entry: It is expressed in Pa.

3.2.4
complex shear modulus
*G**
ratio of the shear stress to the shear strain, where each is a vector which can be represented by a complex number

$$G^* = G' + iG''$$

Note 1 to entry: It is expressed in Pa.

3.2.5
absolute complex shear modulus
|*G**|
absolute value of the *complex shear modulus* (3.2.4)

$$\left|G^*\right| = \sqrt{G'^2 + G''^2}$$

Note 1 to entry: It is expressed in Pa.

3.2.6
elastic normal modulus
storage normal modulus
elastic Young's modulus
E′
component of the applied normal stress, which is in phase with the normal strain, divided by the strain

$$E' = \left|E^*\right| \cos\delta$$

Note 1 to entry: It is expressed in Pa.

3.2.7
loss normal modulus
loss Young's modulus
E″
component of the applied normal stress, which is in quadrature with the normal strain, divided by the strain

$$E'' = \left|E^*\right| \sin\delta$$

Note 1 to entry: It is expressed in Pa.

3.2.8
complex normal modulus
complex Young's modulus
*E**
ratio of the normal stress to the normal strain, where each is a vector which can be represented by a complex number

$$E^* = E' + iE''$$

Note 1 to entry: It is expressed in Pa.

3.2.9
absolute complex normal modulus
absolute value of the *complex normal modulus* (3.2.8)

$$\left|E^*\right| = \sqrt{E'^2 + E''^2}$$

3.2.10
storage spring constant
dynamic spring constant
K'
component of the applied load, which is in phase with the deflection, divided by the deflection

$$K' = \left|K^*\right| \cos\delta$$

Note 1 to entry: It is expressed in N/m.

3.2.11
loss spring constant
K''
component of the applied load, which is in quadrature with the deflection, divided by the deflection

$$K'' = \left|K^*\right| \sin\delta$$

Note 1 to entry: It is expressed in N/m.

3.2.12
complex spring constant
K^*
ratio of the load to the deflection, where each is a vector which can be represented by a complex number

$$K^* = K' + iK''$$

Note 1 to entry: It is expressed in N/m.

3.2.13
absolute complex spring constant
$|K^*|$
absolute value of the *complex spring constant* (3.2.12)

$$\left|K^*\right| = \sqrt{K'^2 + K''^2}$$

Note 1 to entry: It is expressed in N/m.

3.2.14
tangent of the loss angle
$\tan\delta$
ratio of the loss modulus to the elastic modulus

Note 1 to entry: For shear stresses, $\tan\delta = \dfrac{G''}{G'}$ and for normal stresses $\tan\delta = \dfrac{E''}{E'}$.

3.2.15
loss factor
L_f
ratio of the *loss spring constant* (3.2.11) to the *storage spring constant* (3.2.10)

$$L_f = \frac{K''}{K'}$$

3.2.16
loss angle
δ
phase angle between the stress and the strain

Note 1 to entry: It is expressed in rad.

3.3 Other terms applying to periodic motion

3.3.1
logarithmic decrement
Λ
natural (Napierian) logarithm of the ratio between successive amplitudes of the same sign of a damped oscillation

3.3.2
transmissibility
V_τ
ratio of the force transmitted to the force applied

4 Symbols

For the purposes of this document, the following symbols apply.

A	(m^2)	test piece cross-sectional area
a and b	(m)	width or side length of test piece
a_T		Williams, Landel, Ferry (WLF) shift factor
b_T		vertical shift factor
α	(rad)	angle of twist
C_p		heat capacity
γ		strain
γ_0		maximum strain amplitude
γ^*		complex strain
d	(m)	diameter of circular test piece
δ	(rad)	loss angle or phase difference
E	(Pa)	Young's modulus
E_c	(Pa)	effective Young's modulus
E'	(Pa)	elastic normal modulus (storage normal modulus)

E''	(Pa)	loss normal modulus (loss Young's modulus)
E^*	(Pa)	complex normal modulus (complex Young's modulus)
$\|E^*\|$	(Pa)	absolute value of complex normal modulus
F	(N)	load
F_0	(N)	maximum load amplitude
f	(Hz)	frequency
G	(Pa)	Shear modulus
G'	(Pa)	elastic shear modulus (storage shear modulus)
G''	(Pa)	loss shear modulus
G^*	(Pa)	complex shear modulus
$\|G^*\|$	(Pa)	absolute value of complex shear modulus
h	(m)	test piece thickness
K	(N/m)	spring constant
K'	(N/m)	storage spring constant (dynamic spring constant)
K''	(N/m)	loss spring constant
K^*	(N/m)	complex spring constant
$\|K^*\|$	(N/m)	absolute value of complex spring constant
k		numerical factor for shape factor correction
k_1		shape factor in torsion
L_f		loss factor
l	(m)	test piece length or distance between test piece holders
λ		extension ratio
Λ		logarithmic decrement
M'	(Pa)	in-phase or storage modulus
M''	(Pa)	out of phase or loss modulus
M^*	(Pa)	complex modulus
$\|M^*\|$	(Pa)	absolute value of complex modulus
m	(kg)	mass
Q	(N·m)	torque
S		shape factor
T	(K)	temperature (in kelvins)

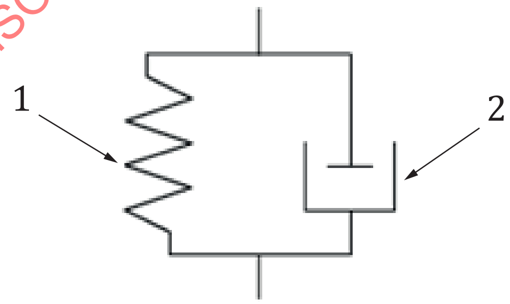
T_g	(K)	low-frequency glass transition temperature
T_0	(K)	reference temperature
t	(s)	time
$\tan\delta$		tangent of the loss angle
τ	(Pa)	stress
τ_0	(Pa)	maximum stress amplitude
τ'	(Pa)	in-phase stress
τ''	(Pa)	out-of-phase stress
τ^*	(Pa)	complex stress
V_τ		transmissibility
ω	(rad/s)	angular frequency
x	(m)	deflection
x_0	(m)	maximum deflection amplitude

5 General

5.1 Viscoelasticity

Matter cannot be deformed without applying force. Unlike elastic materials such as metals, rubber is a viscoelastic material, i.e. it shows both an elastic response and a viscous drag when deformed. Viscoelastic properties have been modelled as combinations of perfectly elastic springs and viscous dampers (dashpots), arranged in parallel (Voigt-Kelvin model) as in Figure 2 or in series (Maxwell model), giving a qualitative model of the time-dependent behaviour of rubber-like materials.

NOTE 1 For the use of more elaborate models to describe the behaviour accurately, see Reference [5].



Key

1 elasticity

2 viscosity

Figure 2 — A dynamic model for rubber (Voigt-Kelvin model)

The dynamic properties of viscoelastic materials can be explained more conveniently by separating the two components elasticity (spring) and viscosity (damping), for example as in Figure 2. Analysis of the behaviour of this model, under a cyclic load or deflection, shows that the resulting deformation or force lags in time behind the applied load or deflection (i.e. shows a phase difference) (see 6.1.1).

NOTE 2 Dynamic properties can be described based on dynamic modulus or dynamic compliance. Both relational expressions are interconvertible. In this document, dynamic modulus is used.

5.2 Use of dynamic test data

Measurements of dynamic properties are generally used for the following purposes:

a) characterization of materials;

b) production of design data;

c) evaluation of products.

Viscoelastic behaviour of rubbers is complex, and the results can be very sensitive to test conditions such as frequency, amplitude of the applied force or deformation, test piece geometry and mode of deformation, so these conditions should be controlled carefully if comparable results are to be obtained.

An important consequence is that it is essential that the conditions under which data are produced are suitable for the intended purpose of the data. In turn, this can mean that different types of test machine can produce test data suitable for different purposes. For instance, small dynamic analyser machines are especially suitable for material characterization but do not necessarily have sufficient capacity for generating design data or measuring product performance.

5.3 Classification of dynamic tests

5.3.1 General

There are numerous types of dynamic test apparatus in use and several ways in which they can be classified, as described in 5.3.2 and 5.3.3.

5.3.2 Classification by type of vibration

There are two basic classes of dynamic test, i.e. free vibration in which the test piece is set in oscillation and the amplitude allowed to decay due to damping in the system, and forced vibration in which the oscillation is maintained by external means (see Table 1). There are two types of test method using forced vibration, i.e. resonance type and non-resonance type.

Table 1 — Classification of dynamic tests by type of vibration

Vibration method	Measuring method	Principle	Pros and cons to be considered
Forced-vibration: The oscillation is maintained by external means.	Non-resonant type	The dynamic properties are calculated based on the amplitude of force and deflection and the phase difference.	The test frequency can be selected arbitrarily from a relatively wide range. Dependency of temperature, frequency and strain amplitude can be measured.
	Resonant type	The dynamic property is determined using the resonance phenomenon at the natural frequency of the system.	This method can be applied up to higher frequency range compared with other methods. It is difficult to distinguish harmonic resonance.

Table 1 *(continued)*

Vibration method	Measuring method	Principle	Pros and cons to be considered
Free-vibration: The oscillation amplitude allowed to decay due to damping.	Free decay	The dynamic properties are obtained from the decay waveform.	The range of test frequency is relatively narrow. Measurements at higher frequencies are difficult. Apparatus is simple and operations are convenient.

5.3.3 Classification by mode of deformation

The deformation method can involve compression, shear, tension or bending of the test piece (see Table 2).

NOTE In the extension state, the cross-sectional area changes due to the extension. The extension load divided by the cross-sectional area is called “true stress”, and that divided by the initial cross-sectional area is called “nominal stress”.

Table 2 — Classification of dynamic tests by deformation modes

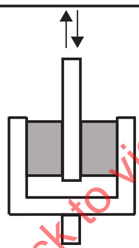
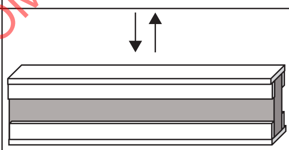
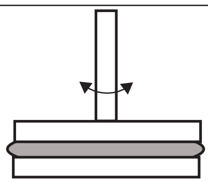
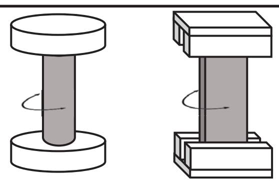
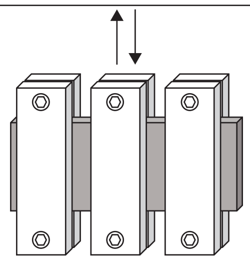
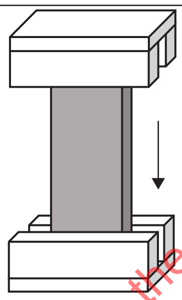
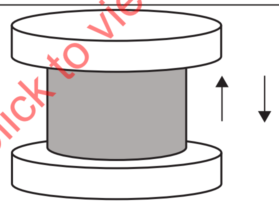
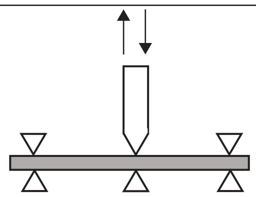
Deformation mode		Direction of movement	Sample shape	Pros and cons to be considered
Shear	Simple shear	Translational	 Test piece shapes are disc or rectangular sheet.	Bonding with adhesive or vulcanization bonding is necessary. Relatively large strains can be applied.
	Pure shear	Translational		The grips must be rigid enough to prevent deformation. The test piece must be adhered or clamped in the grips so that it is held firmly.
	Rotational shear	Rotational		Strain distribution is not uniform. Bonding with adhesive or vulcanization bonding is necessary.

Table 2 *(continued)*

Deformation mode	Direction of movement	Sample shape	Pros and cons to be considered
Torsional shear	Rotational		Preparation of test pieces is simple and convenient. Measurement with large deformation is difficult.
Film shear	Translational		Preparation and mounting of test pieces are simple and convenient. Crimping with bolts or bonding with adhesive is necessary. In large deformation, the test piece can slip between the clamps compared with bonding methods.
Tension	Translational	 Static tension is needed	Nominal stress is applied (see NOTE). Preparation of test pieces is simple and convenient. Strain distribution is relatively uniform.
Compression	Translational	 Static compression is needed	Relatively large force is needed.
Bending	Translational		This method is usually applied to relatively stiff and inextensible materials such as rubber/fibre composite materials.

5.4 Factors affecting machine selection

The advantages and disadvantages of the various types of dynamic test machine can be summarized as follows.

Deformation in simple shear generally allows the most precise definition of strain, and the stress-strain curve remains linear to higher amplitudes than for other deformation modes, but the test pieces have to be fabricated with metal end pieces.

Deformation in compression can be useful in matching service conditions, particularly with products, but generally requires a higher force capacity and consideration of the shape factor of the test piece.

Deformation in bending, torsion or tension requires a lower force capacity and test pieces are easily produced, but it can be less satisfactory for measurements of absolute values of the modulus.

The preferred type of test machine for generating design data is a forced-vibration non-resonance machine operating in shear.

A large force capacity, and hence an expensive machine, is necessary for higher strain amplitudes in shear and compression and for testing products.

For material characterization, the mode of deformation is not, in principle, important and a large force capacity is not necessary.

Dynamic analysers of modest capacity but having automated scanning of frequency and temperature are particularly efficient for material characterization.

Free-vibration apparatus is restricted to low frequencies and amplitudes, normally in torsion.

In tension and compression modes, a static strain which is larger than the dynamic strain intended to be tested is necessary.

When an adhesive is used to prevent slip between the test pieces and the holders, special care should be taken to eliminate the effect of the adhesive.

In the case of a temperature ramp measurement, it is preferable to have a mechanism to compensate for the influence of thermal expansion or shrinkage of the test piece.

With large deformation, necking (in tension mode) or buckling (in torsion mode) can occur.

6 Principles of dynamic motion for each vibration method

6.1 Forced vibration non-resonant method

6.1.1 Dynamic motion in linear response

Rubbers are viscoelastic materials, and hence their response to dynamic excitation is a combination of an elastic response and a viscous response and energy is lost in each cycle.

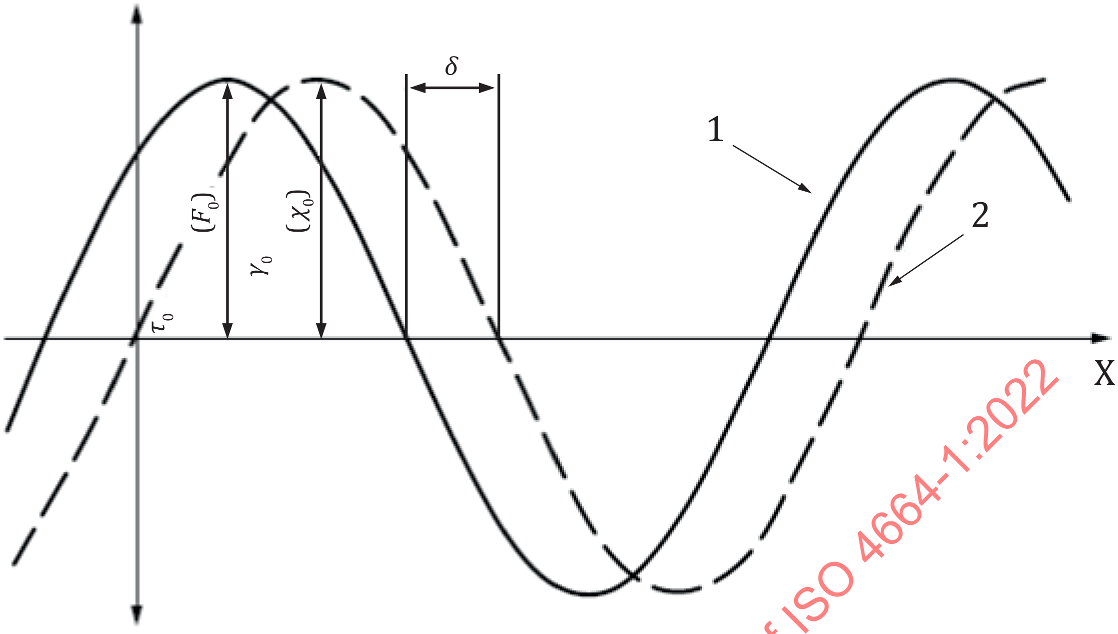
For sinusoidal strain, the motion is described by Formula (1):

$$\gamma = \gamma_0 \sin \omega t \tag{1}$$

where

- γ is the strain;
- γ_0 is the maximum strain amplitude;
- ω is the angular frequency;
- t is the time.

See Figure 3.



Key

X ωt

1 stress (load)

2 strain (deflection)

Figure 3 — Sinusoidal stress-strain time cycle

The stress τ will not be in phase with the strain and can be considered to precede it by the phase angle δ so that Formula (2) applies:

$$\tau = \tau_0 \sin(\omega t + \delta) \tag{2}$$

where

τ is the stress;

τ_0 is the maximum stress amplitude;

δ is the phase or loss angle.

In the case of tensile or compressive deformation mode, a dynamic sinusoidal strain or stress is applied on a static strain or static stress. The static strain or stress is referred to as “mean strain” or “mean stress”, and the maximum dynamic strain or stress amplitude is measured from the mean strain or mean stress.

Considering the stress as a vector having two components, one in phase (τ') and the other 90° out of phase (τ''), and defining the corresponding in-phase modulus (storage modulus) as M' and the corresponding out-of-phase modulus (loss modulus) as M'', the complex modulus (M^*) is given by Formula (3):

$$M^* = M' + iM'' \tag{3}$$

Also Formulae (4) and (5):

$$M' = \frac{\tau'}{\gamma_0} = \frac{\tau_0}{\gamma_0}\cos\delta = \left|M^*\right|\cos\delta \tag{4}$$

$$M'' = \frac{\tau''}{\gamma_0} = \frac{\tau_0}{\gamma_0}\sin\delta = \left|M^*\right|\sin\delta \tag{5}$$

The absolute value of the complex modulus is given by Formula (6):

$$\left|M^*\right| = \sqrt{M'^2 + M''^2} \tag{6}$$

The tangent of the loss angle is given by Formula (7):

$$\tan\delta = \frac{M''}{M'} \tag{7}$$

where

M^*	is the complex modulus;
M'	is the elastic modulus;
M''	is the loss modulus;
$\lvert M^*\rvert$	is the absolute complex modulus;
$\tan\delta$	is the tangent of loss angle.

The moduli are named according to the deformation mode, see Table 3.

Table 3 — Different kinds of moduli and their deformation modes

Kind of modulus	Shear mode	Tension mode
Complex modulus	Complex shear modulus	Complex normal modulus Complex Young's modulus
Elastic modulus	Elastic shear modulus Storage shear modulus	Elastic normal modulus Storage normal modulus Elastic Young's modulus
Loss modulus	Loss shear modulus	Loss normal modulus Loss Young's modulus
Absolute complex modulus	Absolute complex shear modulus	Absolute complex normal modulus

6.1.2 Dynamic motion with nonlinear response

In general, the stress-strain behaviour of viscoelastic materials exhibits nonlinearity with increasing strain or stress. The response stress wave corresponding to the sinusoidal strain will include third, fifth or higher harmonic waves. In the case of sufficiently small strain, the measurements are made under linear conditions, and high harmonics components are negligible. However, the harmonics increase with increasing strain amplitude, and the response stress shows nonlinear behaviour. When the degree of nonlinearity (see Annex A) is 3 % or less, the harmonic components can be neglected. Alternatively, the nonlinearity can be estimated by distortion of the hysteresis loop (see Annex A).

When nonlinearity is present, it appears as distortion of the hysteresis loop from a pure ellipse. When the strain is small, the hysteresis loop is an almost perfect ellipse, and the phase difference can be determined from its area, but the area obtained from a hysteresis loop with large strain is an apparent phase difference (average phase difference), and has no physical meaning (see Annex A).

6.1.3 Free-vibration method

For a freely vibrating rubber test piece and mass system, the motion is described by Formula (8):

$$m\frac{d^2x}{dt^2}+\frac{K''}{\omega}\frac{dx}{dt}+K'x=0 \tag{8}$$

where

K'	is the storage spring constant (N/m);
K''	is the loss spring constant (N/m);
m	is the mass (kg);
x	is the displacement (m);
ω	is the angular frequency (rad/s).

The natural logarithm of the ratio between successive amplitudes is called the “logarithmic decrement”, and is obtained according to Formula (9):

$$\Lambda=\log_e\left(\frac{x_n}{x_{n+1}}\right) \tag{9}$$

where

Λ	is the logarithmic decrement;
n	is the number of the cycle;
x_n	is the amplitude of the nth cycle (m);
x_{n+1}	is the amplitude of the (n+1)th cycle (m).

The storage spring constant, the loss spring constant and the loss factor are determined by Formulae (10) to (12) using the logarithmic decrement. The angular frequency ω is obtained from the oscillation period t_c with $\omega = 2\pi/t_c$.

$$K'=m\omega^2\left(1+\frac{\Lambda^2}{4\pi^2}\right) \tag{10}$$

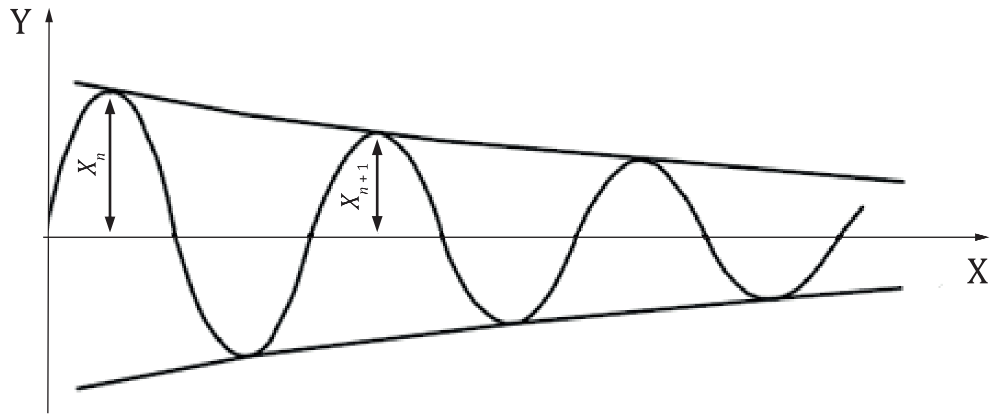
$$K''=\frac{m\omega^2\Lambda}{\pi} \tag{11}$$

$$L_f=\frac{\Lambda}{\pi\left(1+\frac{\Lambda^2}{4\pi^2}\right)} \tag{12}$$

where

Λ	is the logarithmic decrement;
L_f	is the loss factor.

See Figure 4.



Key

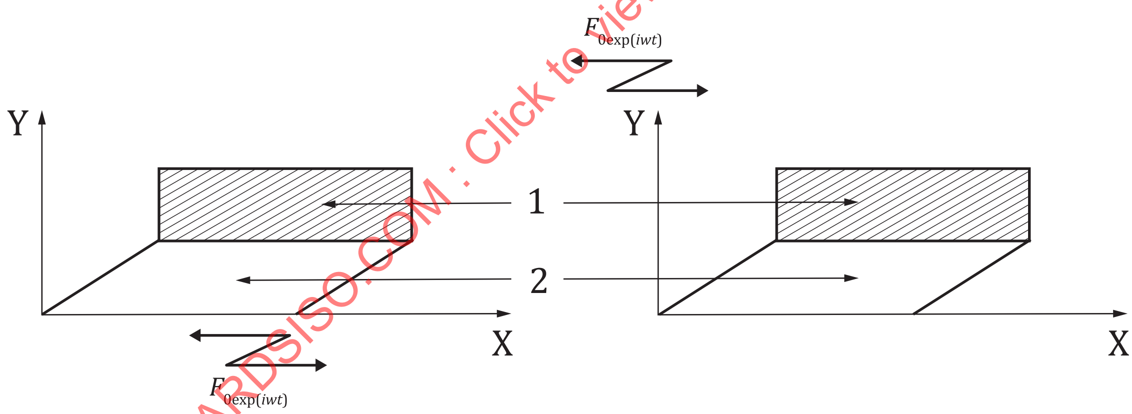
X time

Y amplitude

Figure 4 — Waveform for free-vibration method

6.2 Forced resonant vibration

The test piece with an added mass is vibrated with increasing frequency and the amplitude of the resulting movement is monitored (see Figure 5). The resonance frequency is that when the amplitude reaches a maximum. The elastic modulus and the loss factor can be determined from the resonant frequency and the resonant amplitude or the resonant magnification.



a) External displacement **b) External force**

Key

1 mass

2 rubber

Figure 5 — Motion excitation and force excitation, external source of oscillation

When a deformation of $\zeta(t) = \zeta_0 \exp(iwt)$ is applied to the support end of the test piece, the equation of motion is expressed as Formula (13):

$$m\frac{d^2x}{dt^2} + K^*[x - \zeta(t)] = 0 \tag{13}$$

where

K^* is the complex spring constant;

$\zeta(t)$ is the deformation;

m is the mass.

By substituting $x = x_0 \exp(i\omega t)$ into the equation and solving, resonance magnification $\mu = \frac{\sqrt{1+L_f^2}}{L_f}$ can be derived.

The storage spring constant and the loss factor are given by Formulae (14) and (15) with the resonant frequency ω_r and the resonance magnification μ.

$$K' = m\omega_r^2 \tag{14}$$

$$L_f = \frac{1}{\sqrt{\mu^2 - 1}} \tag{15}$$

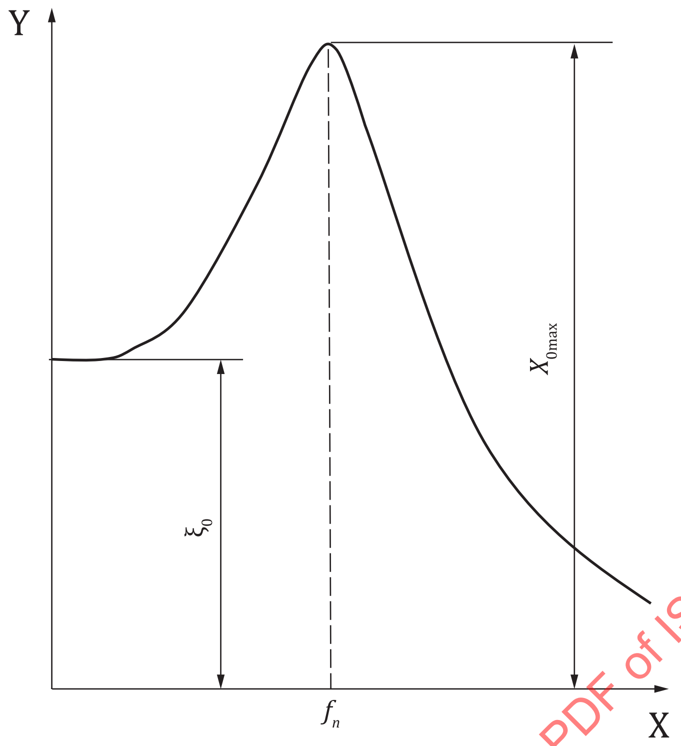
where

K' is the dynamic (or storage) spring constant;

μ is the resonance magnification;

ω_r is resonant frequency;

L_f is the loss factor.



Key

X frequency, f

Y relative amplitude, x_0

Figure 6 — Example of resonance curve in forced resonant method

The transmissibility V_τ is given by Formula (16):

$$V_\tau = \sqrt{\frac{1+L_f^2}{\left[1-\left(\frac{\omega}{\omega_n}\right)^2\right]^2 + L_f^2}} \tag{16}$$

7 Test parameter dependence

7.1 Interdependence of frequency and temperature (time – temperature superposition)

Measuring the temperature dependency of rubber materials is useful for analysing the viscosity change with temperature or the thermal transitions. Phase transitions can be detected and analysed to locate components in polymer blends. Impact properties, crystallinity and other morphological properties can be derived from these experiments.

The effects of frequency and temperature are interdependent, i.e. an increase in temperature can produce a similar change in modulus as a reduction in frequency, and vice versa. This can be used to make estimates of dynamic properties outside the measured range, for example at higher frequencies than an apparatus can achieve, by using results at lower temperatures.

A master curve of a viscoelastic characteristic over a wide frequency range can be obtained by horizontally moving a frequency dependence curve measured at various temperatures along the frequency axis (see Annex B).

The amount of lateral movement of each test temperature with respect to a reference temperature is called a “shift factor”. The shift factor a_T for compounded rubbers is often represented by the WLF equation as shown by Formula (17):

$$\log_{10}[a_T] = \frac{-c_1(T-T_0)}{c_2+(T-T_0)} \tag{17}$$

c_1 and c_2 are coefficients depending on the rubber material and the reference temperature, but when the reference temperature is set as the glass transition temperature T_g, it has been shown that the simple equation given in Formula (18) can be applicable for many rubber materials:

$$\log_{10}[a_T] = \frac{-17{,}44\left(T-T_g\right)}{51{,}6+\left(T-T_g\right)} \tag{18}$$

where T_g is the low-frequency (dilatometric) glass transition temperature.

Many refinements to the general procedures outlined here have been developed. Limitations arise especially due to fillers or crystalline zones, and care should be taken in applying the temperature/frequency transformation. It can be well suited to describing the large variations in a property observed when the temperature and frequency cover wide ranges, but is less applicable to the transformation of data obtained over limited ranges. Transformations greater than one decade from the measured data become less reliable.

7.2 Strain amplitude

Measurement of the dependence of modulus on strain amplitude can be used to determine the range of linear viscoelastic behaviour of a material. In the viscoelastic region, the modulus is independent of strain amplitude and is said to be linear viscoelastic, but above a certain amplitude the modulus drops with increasing strain due to the break-down of the internal structure of the material. This phenomenon is called the “Payne effect”[6][7].

8 Conditioning

8.1 Storage

The time lapse between vulcanization and testing should be in accordance with ISO 23529.

8.2 Temperature

Test pieces should be conditioned at a standard laboratory temperature for not less than 3 h immediately before a sequence of tests. At each temperature, it is essential that the test piece is conditioned for sufficient time to reach equilibrium, but conditioning should be no longer than is necessary, particularly at higher temperatures, to avoid ageing effects. The conditioning time depends on the test piece dimensions and the temperature. Guidance is given in ISO 23529.

8.3 Mechanical conditioning

Dynamic properties of filled rubbers are dependent on their strain history and temperature history, and it is necessary to pre-condition the test pieces to obtain consistent and reproducible results.

The test pieces should be mechanically conditioned before being tested (sometimes referred to as “scragging”) to remove irreversible “structure”. The conditioning should consist of at least six cycles at the maximum strain and temperature to be used in the test series.

A minimum of 12 h is recommended between mechanical conditioning and testing to allow reversible “structure” to equilibrate.

Where the dynamic test is to be superimposed on a static pre-strain, the test piece should be held at the static strain during the rest period.

This mechanical conditioning can generally be omitted when only a single, very small, strain is used as, for example, in free vibration. This omission should be mentioned in the test report.

When the tests are intended to characterize the strain dependency of the material, the mechanical conditioning can be omitted so that Payne effect can be clearly shown.

9 Forced vibration non-resonant method

9.1 Apparatus

Both translational and rotational modes of test piece deformation can be used in forced vibration non-resonant methods.

All methods require the following basic elements.

9.1.1 Clamping or supporting arrangement that permits the test piece to be held so that it acts as the elastic and viscous element in a mechanically oscillating system.

9.1.2 Device for applying an oscillatory load (stress) or torque to the test piece.

9.1.3 Detectors, for determining dependent and independent experimental parameters such as force (or torque), displacement, frequency and temperature.

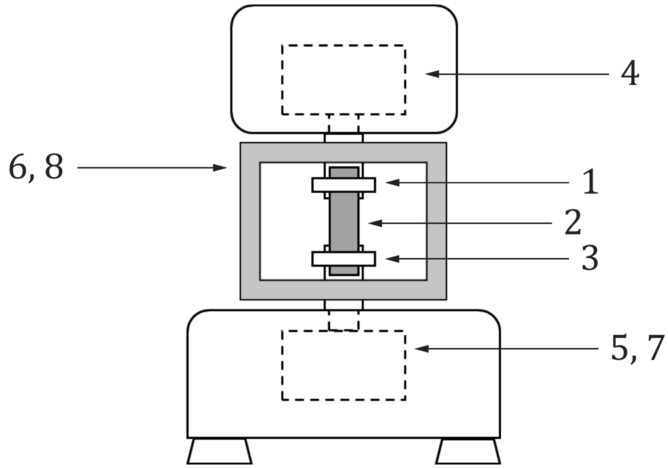
9.1.4 Data acquisition and processing system: Data-processing equipment should be capable of recording the signals of force and displacement cycle amplitudes.

9.1.5 Oven and controller, for maintaining the test piece at the required temperature. It is recommended that the chamber be equipped with temperature programming facilities. The temperature in the oven should be uniform within ±1 °C.

9.1.6 Temperature-measurement device, for measuring the temperature of the air surrounding the test piece should be capable of determining the temperature to ±0,1 °C.

9.1.7 Instruments for measuring test piece dimensions, in accordance with ISO 23529.

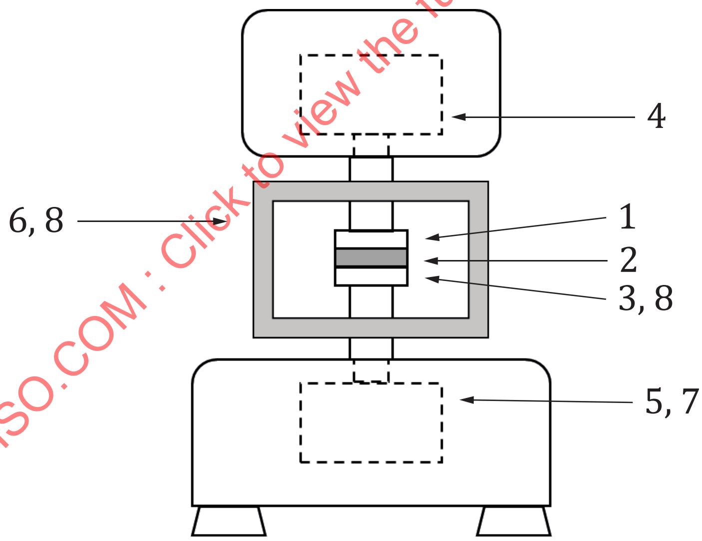
Numerous forms of test machine have been developed and used successfully both by individual experimenters and commercial manufacturers. Figures 7 and 8 give schematic illustrations for typical examples of machines that have translational motion and rotational motion for deformation of the test piece, respectively.



Key

1 upper test piece holder (geometry)
2 test piece
3 lower test piece holder (geometry)
4 force transducer
5 displacement detector
6 temperature controlled chamber
7 linear drive motor
8 thermometer

Figure 7 — Example of translational motion test apparatus



Key

1 upper test piece holder (geometry)
2 test piece
3 lower test piece holder (geometry)
4 torque transducer
5 displacement detector
6 temperature controlled chamber
7 vibrator
8 thermometer

Figure 8 — Example of rotational motion test apparatus

9.2 Test piece

9.2.1 Test piece preparation

Test pieces can be moulded or cut from moulded sheet. Moulding is preferred for shear and compression test pieces. Metal plates for shear and compression test pieces can be bonded during moulding or bonded afterwards with a thin layer of suitable adhesive.

Test pieces can be obtained from some products by cutting and buffing. In other cases, it can be necessary or desired to test the complete product.

9.2.2 Test piece shapes and dimensions

Test piece shapes and dimensions will vary according to the mode of deformation, the type of test machine and its capacity (see Table 4).

The thickness of any plates that are bonded to the rubber during vulcanization should be measured before moulding and the thickness of the rubber deduced by measurement of the overall thickness of the moulding.

To study practical performance, test pieces of shape and dimensions other than described in Table 4 can be tested.

The dimensions of the test pieces should be determined in accordance with ISO 23529.

Table 4 — Test pieces in various deformation modes for forced vibration non-resonant methods

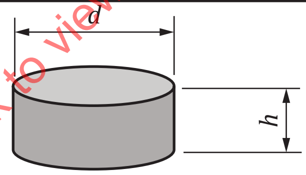
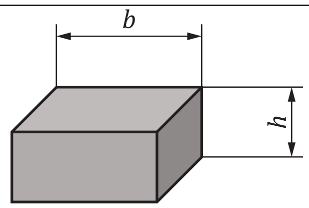
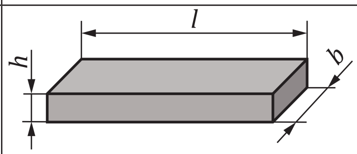
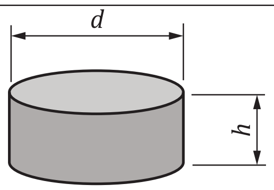
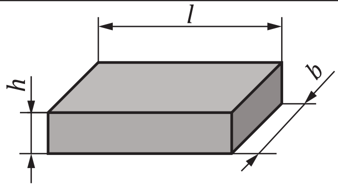
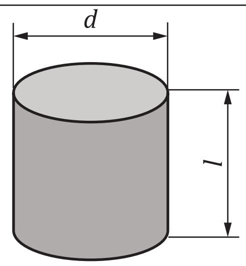
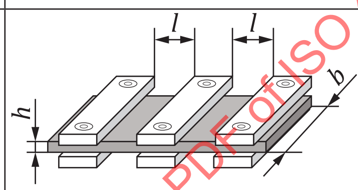
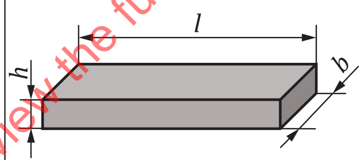
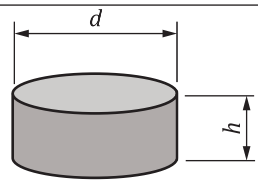
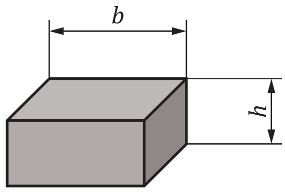
Deformation mode		Dimensions	Test piece shapes	Comments
Shear	Simple shear	disc or cylinder $h \leq 12$ mm $d/h \geq 4$		In the case of $d/h < 4$ or $b/h < 4$, the deformation includes a tension component. It is then necessary to treat the result with caution.
		rectangular column $h \leq 12$ mm $b/h \geq 4$		
	Pure shear	rectangular strip h: 0,3 mm to 2 mm $b \geq 5$ mm (distance between grips) $l \geq 50$ mm $l/b \geq 10$		The width of the test piece shall be at least 10 times the distance between the grips.
	Rotational shear	disc or cylinder h: 0,5 mm to 6 mm d: 8,5 mm to 25 mm		The test piece is placed on the centre of the holders.

Table 4 *(continued)*

Deformation mode	Dimensions	Test piece shapes	Comments
Torsional shear	rectangular strip h: 0,3 mm to 6 mm b: 5 mm to 13 mm l: 5 mm to 40 mm (distance between clamps)		The end portions of the test piece should be of sufficient length to ensure adequate gripping by the clamps.
	disc or cylinder d: 1,5 mm to 4,5 mm l: 5 mm to 40 mm (distance between clamps)		
Film shear	rectangular sheet h: 0,1 mm to 4 mm b: more than 20 mm $b/l \geq 4$		In the case of $b/l < 4$, the deformation includes a bending component.
Tension	rectangular strip h: 0,1 mm to 4 mm b: 2 mm to 12 mm l: 5 mm to 60 mm (distance between clamps) l/b: 2,5 to 5		The end portions of the test piece should be of sufficient length to ensure adequate gripping by the clamps.
Compression	disc $d/h \leq 1,5$ h: 1 mm to 10 mm rectangular column $h \leq 12$ mm $b/h \geq 4$ the ISO 815-1 test piece with d = 29,0 mm h = 12,5 mm is suitable. A rectangular section test piece can also be used.	  The disc shape is preferable.	Both end faces should be parallel and smooth.
Bending	rectangular strip h: 1 mm to 3 mm b: 4 mm to 12 mm $l/h \geq 16$		

9.2.3 Number of test pieces

In order to obtain an indication of the variability of the material, it is recommended that a minimum of three test pieces or products be tested. However, for routine and screening tests, the number of test pieces can be reduced to one or two.

9.3 Test conditions

9.3.1 Strain

Rubbers containing substantial quantities of fillers show viscoelastic behaviour that is dependent on the strain amplitude of the test. As a general principle, strain amplitudes should be chosen to correspond to the strains experienced in service but, in practice, there can be restrictions because of machine capacity, the wish to operate in the linear part of the stress-strain curve and heat build-up.

Recommended values of strain amplitude are given in Table 5. Not all of these strain amplitudes will necessarily be required for a given series of tests.

In practice, the lowest strain level achievable will be limited by machine sensitivity, and the highest strain level by the machine capacity, especially at higher frequencies and at temperatures near the glass transition.

In service, products can be subjected to a dynamic strain superimposed on a static strain, and the static strain does not necessarily have the same mode of deformation. To obtain data more relevant to such conditions, the dynamic strains recommended here can be superimposed on any level or form of static strain. This can be particularly relevant to testing products and is usually applied to compression test pieces.

In certain cases, a material can be characterized better by conducting the tests under an applied stress. In such cases, the test conditions should be agreed between the interested parties.

When testing with a large strain amplitude or high frequency, there is an increasing danger of heat build-up in the test piece, and the test time should be as short as possible, especially at or near transitions. The temperature rise can be estimated as follows.

The energy loss per unit volume per cycle, that is the power loss, is as given by Formula (19):

$$\pi \cdot \sin \delta \left(\left| M^* \right| \cdot \gamma_0^2 \right) \tag{19}$$

Thus, the rate of rise in temperature when there are no heat losses from the test piece is as given by Formula (20):

$$\Delta T = \pi \cdot \sin \delta \left(\frac{\left| M^* \right| \cdot \gamma_0^2 \cdot f}{C_p} \right) \tag{20}$$

where C_p is the heat capacity per unit volume [a typical value is 1,7 MJ/(m^{3}K)].

9.3.2 Frequency and temperature

Rubbers show viscoelastic behaviour which is frequency and temperature dependent. This dependence is very marked near transitions. Consequently, frequencies and temperatures relevant to service should be chosen or, particularly when characterizing materials, tests should be carried out over a range of frequencies and temperatures.

Recommended values are given in Table 5.

Table 5 — Recommended test conditions for various deformation modes

Deformation mode		Strain	Temperature	Frequency
Shear	Simple shear	Dynamic strain: 0,05 % to 50 % Static strain: 0 %	Range: −120 °C to 120 °C Tolerance: ±1 °C	Range: 0,1 Hz to 200 Hz Tolerance: ±2 %
	Pure shear	Dynamic strain: 0,1 % to 10 % Static strain should be more than 1,2 times the dynamic strain.		
	Rotational shear	Dynamic strain: 0,05 % to 50 % Static strain: 0 %		
	Torsional shear	Dynamic strain: 0,05 % to 15 % Static strain: 0 %		
	Film shear	Dynamic strain: 0,05 % to 50 % Static strain: 0 %		
Tension		Dynamic strain: 0,1 % to 10 % Static strain should be more than 1,2 times the dynamic strain.		
Compression		Dynamic strain: 0,1 % to 5 % Static strain should be more than 1,2 times of the dynamic strain.		
Bending		Dynamic strain: 0,1 % to 5 %		

9.4 Test procedure

When measurements are carried out over a range of test conditions, in principle, it is preferable to conduct them in the following order (see Table 6).

If a test piece is to be tested under more than one set of conditions, measurements should begin with the least severe conditions and then proceed to larger amplitudes and higher frequencies. In the case of testing at different temperatures, the test chamber should be adjusted to the lowest specified temperature and, after the test pieces have tested at that temperature, the chamber should be raised to the next temperature required.

For forced-vibration tests, measurement should be made after at least six cycles have been applied to reach near-equilibrium and should be conducted with increasing frequency. At low amplitudes and frequencies, there is no need to restrict the length of time for which cycling is continued. However, at higher amplitudes and frequencies there is an increasing danger of heat build-up in the test piece, and the test time should be as short as possible, especially at or near transitions.

Tests should be conducted with increasing test temperature, either in steps or continuously at a rate such that the test piece can reach equilibrium temperature. In the case of large test pieces, special attention should be paid to obtain uniform temperature in whole test piece. The strain amplitude can be different in the transition and rubbery regions.

At the same mean strain (static strain), tests should be conducted with increasing strain amplitude.

Table 6 — Recommended test condition for programmed scan

Parameter	Rate of condition change	Measuring interval
Temperature scan	From low to high temperature Rate of temperature rising Continuous: 0,2 °C to 5 °C /min Stepwise: hold for 2 min at each temperature increase 1 °C /min to 5 °C /min between each temperature	Temperature interval: less than 2 °C. In the transition range, a smaller interval is preferred.
Frequency scan	From low to high frequency	Frequency interval: less than 1 decade. Logarithmic equal interval is preferred.
Strain scan	From small to large strain	Strain interval: less than 1 decade. Logarithmic equal interval is preferred.

9.5 Expression of results

9.5.1 Parameters required

Generally, the in-phase, out-of-phase and complex moduli (or complex spring constants) and tanδ (or L_f) are required. Where appropriate, these are best presented in tables or graphically as a function of temperature, frequency and amplitude.

9.5.2 Wave-form method

The load and deflection waveforms are recorded separately, and the maximum load amplitude (F_0), the maximum deflection amplitude (x_0) and the phase difference (Δt) between the load and deflection are measured. The loss angle is determined with the time of one cycle (t_c) and the phase difference (Δt) from Formula (21):

$$\delta = 2\pi\left(\frac{\Delta t}{t_c}\right) \tag{21}$$

NOTE Alternatively, the load and deflection curves are subjected to Fourier transforms, and the loss angle δ can be determined from the time shift between the two curves.

The absolute complex spring constant is given by Formula (22):

$$\left|K^*\right| = {F_0}/{x_0} \tag{22}$$

The storage and loss spring constants and loss factor are calculated with Formulae (23) to (25):

$$K' = \frac{F'}{x_0} = \frac{F_0}{x_0}\cos\delta = \left|K^*\right|\cos\delta \tag{23}$$

$$K'' = \frac{F''}{x_0} = \frac{F_0}{x_0}\sin\delta = \left|K^*\right|\sin\delta \tag{24}$$

$$L_f = {K''}/{K'} = \tan\delta \tag{25}$$

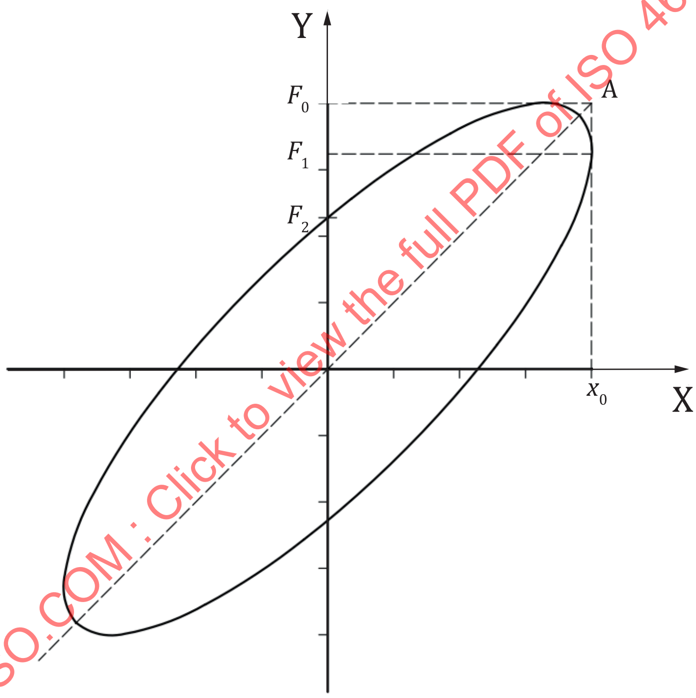
This method should not be applied to a material that has nonlinear dynamic properties. The Fourier transform method allows analysis of nonlinear dynamic forces in a manner that minimizes the influence of force wave shape. A popular algorithm for the transform is the fast Fourier transform (FFT). In this method, both the dynamic motion and force signals are digitized and then subjected to

Fourier analysis. Through the transform, the fundamental and harmonic components of each waveform are calculated. The fundamental component is that having the same frequency as the imposed motion. The fundamental component of both the dynamic motion and force can be used for the calculation with the formulae above.

9.5.3 Hysteresis loop method

The parameters can be derived from the load-deflection curve, an example of which is shown in Figure 9. This can be achieved by suitable electronic analysis techniques without the need to record the load-deflection loop. However, with filled rubbers and at higher amplitudes, nonlinear behaviour can be exhibited and the hysteresis loop will deviate from a perfect ellipse, which complicates the derivation of the parameters.

Figure 9 shows a load-deflection loop obtained from a dynamic test on a double-shear test piece. The origin O represents the mean values of the load and deflection and, if a static deflection is imposed, will not be the zero values. The loads and deflections shown are thus the dynamic components.



Key

X deflection

Y load

Figure 9 — Load-deflection curve

If the behaviour of the rubber is linear, the loop shown in Figure 9 will be an ellipse. In this case, for the double-shear test piece, the absolute complex shear modulus is given by Formula (26):

$$\left|G^*\right| = \frac{F_0 h}{2Ax_0} \tag{26}$$

where

F_0 and x_0 are the maximum load amplitude and maximum deflection amplitude, respectively;

A is the test piece cross-sectional area (m^2);

h is the test piece thickness (m).

Thus, F_0/x_0 is given (see Figure 9) by the slope of the line OA which is the diagonal of the circumscribed rectangle. The loss angle is given by Formula (27):

$$\tan\delta = \frac{F_2}{F_1} \tag{27}$$

The elastic shear modulus G′ is given by Formula (28):

$$G' = \left|G^*\right|\cos\delta = \frac{h}{2A}\frac{F_1}{x_0} \tag{28}$$

and the loss shear modulus G″ by Formula (29):

$$G'' = \left|G^*\right|\sin\delta = \frac{h}{2A}\frac{F_2}{x_0} \tag{29}$$

The loss angle is also given from the area of the hysteresis loop Δw by Formula (30):

$$\sin\delta = \frac{\Delta w}{\pi F_0 x_0} \tag{30}$$

This latter relation can be particularly useful when there is some nonlinearity and the ellipse is not perfect, as it will give an average value.

Similar expressions apply to other modes of deformation and other test piece geometries.

9.5.4 Stress-strain relationships and shape factors

In shear, stress can be taken as proportional to strain as given in Formula (31):

$$\tau = G\gamma \tag{31}$$

With the recommended test piece, no shape factor correction is necessary.

In tension or compression, the stress-strain relationship is better represented by Formula (32):

$$\tau = \frac{E}{3}\left(\lambda - \lambda^{-2}\right) \tag{32}$$

where

τ is the stress with reference to the initial cross-section (Pa);

E is Young's modulus (Pa);

λ is the extension ratio.

For compression test pieces with bonded ends, a shape factor needs to be applied (see ISO 7743:2017, Annex B), as shown by Formula (33):

$$E_c = E\left(1 + 2kS^2\right) \tag{33}$$

where

E_c is the effective Young's modulus (Pa);

k is a numerical factor;

S is the shape factor in compression.

NOTE S is given by:

$$S = \frac{\pi \left(d/2 \right)^2}{\pi d h} = \frac{d}{4h} \text{ (for disc or cylindrical test pieces)}$$

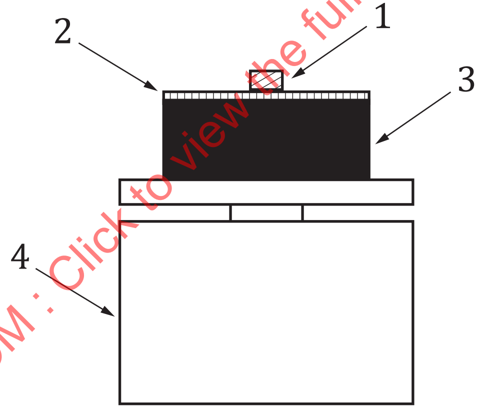
$$S = \frac{ab}{2h(a+b)} \text{ (for rectangular column test pieces)}$$

Because the relationship between dynamic stiffness and basic modulus can be complex and only approximate, it can be preferable, particularly for products, to work in stiffness.

10 Forced vibration resonant method

10.1 Apparatus

The test apparatus consists of a vibrator, detectors for oscillation amplitude and force, and a data acquisition and processing system, as shown in Figure 10.



key

1 sensor

2 mass

3 test piece

4 vibrator

Figure 10 — Example of apparatus for forced vibration resonant method

10.2 Expression of results

One end of the test piece is oscillated at a specified amplitude ξ_0 while changing the frequency f, and the amplitude x_0 of the mass m added to the other end is measured, and a resonance curve is recorded. From the resonance curve, the resonance frequency f_n at which x_0 reaches maximum, and the maximum amplitude $x_{0\max}$ of the additional mass m are determined. From these values, the resonance magnification μ is obtained and the elastic modulus and the loss factor are calculated using Formulae (34), (35), (36) and (37):

$$\mu = \frac{x_{0\max}}{\zeta_0} \tag{34}$$

$$K' = 4\pi^2 m f_n^2 \tag{35}$$

$$L_f = \frac{1}{\sqrt{\mu^2 - 1}} = \tan\delta \tag{36}$$

$$K'' = K' \cdot L_f \tag{37}$$

where

μ is the resonance magnification;

x_0 is the amplitude of the mass m;

ξ_0 is the oscillation amplitude;

f_n is the resonance frequency, $\omega_n = 2\pi f_n$;

K' is the storage spring constant (N/mm) or (Nm/rad);

K'' is the loss spring constant (N/mm) or (Nm/rad);

L_f is the loss factor.

It is preferable to search for the resonance point in advance with an amplitude smaller than the test amplitude so that the test time can be shortened.

11 Free-vibration method

11.1 General

The basic principles of dynamic testing using the free-vibration method are accordance with ISO 4664-2 and as given in 11.2 and 11.3.

11.2 Test piece dimensions

Rectangular strips of thickness between 1 mm and 3 mm of width between 4 mm and 12 mm (subject to a maximum width to thickness ratio of 10) and of length between the clamps at least 10 times the width (subject to a maximum of 120 mm) are preferred. The thickness, width and distance between grips shall be measured to ±1 %.

11.3 Test conditions

Strain amplitude	0,5 % max.
Frequency	0,1 Hz to 10 Hz.
Temperature	Continuous scans of properties against temperature may be obtained or temperatures selected from ISO 23529.