INTERNATIONAL STANDARD

ISO 4664

Third edition
1998-05-01

Rubber — Guide to the determination of dynamic properties

Caoutchouc — Lignes directrices pour la détermination des propriétés dynamiques

Foreword

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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.

International Standard ISO 4664 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

This third edition cancels and replaces the second edition (ISO 4664:1987) as well as ISO 2856:1981, of which it constitutes a technical revision.

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Printed in Switzerland

Rubber — Guide to the determination of dynamic properties

WARNING – Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard provides guidance on the determination of dynamic properties of vulcanized and thermoplastic rubbers. It includes both free- and forced-vibration methods for use with both materials and products. It does not cover rebound resilience nor cyclic tests in which the main objective is to fatigue the rubber.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 471:1995, *Rubber – Temperatures, humidities and times for conditioning and testing.*

ISO 815:1991, *Rubber, vulcanized or thermoplastic – Determination of compression set at ambient, elevated or low temperatures.*

ISO 3383:1985, *Rubber – General directions for achieving elevated or subnormal temperatures for test purposes.*

ISO 4648:1991, *Rubber, vulcanized or thermoplastic – Determination of dimensions of test pieces and products for test purposes.*

ISO 4663:1986, *Rubber – Determination of dynamic behaviour of vulcanizates at low frequencies – Torsion pendulum method.*

ISO 5893:1993, *Rubber and plastics test equipment – Tensile, flexural and compression types (constant rate of traverse) – Description.*

ISO 7743:1989, *Rubber, vulcanized or thermoplastic – Determination of compression stress-strain properties.*

3 Definitions

For the purposes of this International Standard, the following definitions apply (for the symbols used, see clause 4):

3.1 Terms applying to any periodic deformation

3.1.1 mechanical-hysteresis loop

The closed curve representing successive stress-strain states of the material during a cyclic deformation.

NOTE – Loops may be centred on the origin of the coordinate system or, more frequently, displaced to various levels of strain or stress; in the latter case, the shape of the loop becomes asymmetrical, but this fact is frequently disregarded.

3.1.2 energy loss (J/m^3)

The energy per unit volume which is lost in each deformation cycle. It is the hysteresis loop area, calculated with reference to coordinate scales.

3.1.3 power loss (W/m^3)

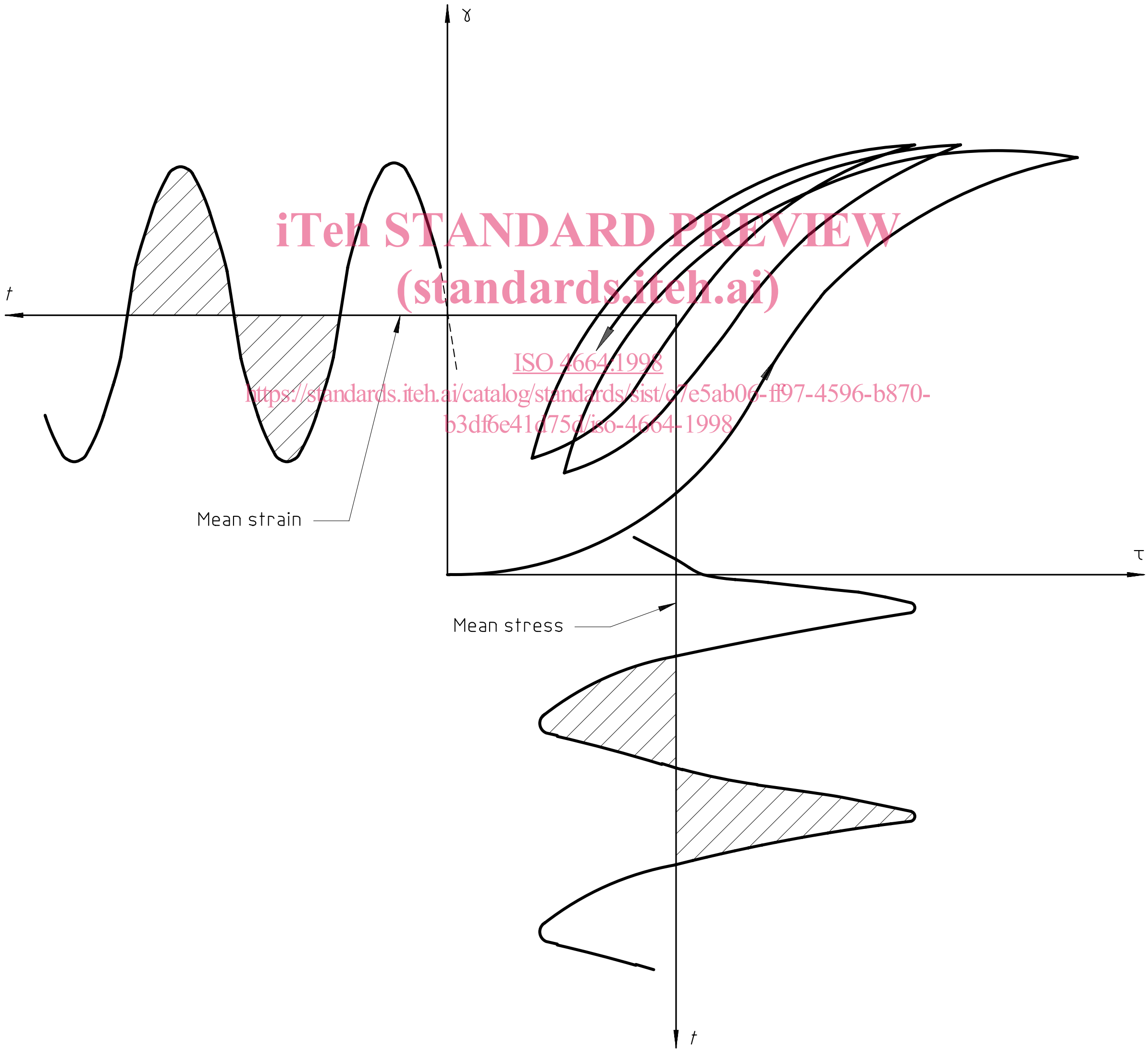
The power per unit volume which is transformed into heat through hysteresis. It is the product of energy loss and frequency.

3.1.4 mean stress (Pa)

The average value of the stress during a single complete hysteresis loop (see figure 1).

3.1.5 mean strain (dimensionless)

The average value of the strain during a single complete hysteresis loop (see figure 1).



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

3.1.6 mean modulus (Pa)

The ratio of the mean stress to the mean strain.

3.1.7 stress amplitude (Pa)

The 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).

3.1.8 root-mean-square stress (Pa)

The square root of the mean value of the square of the stress averaged over one deformation cycle.

NOTE – For a symmetrical sinusoidal stress, the root-mean-square stress equals the stress amplitude divided by $\sqrt{2}$.

3.1.9 strain amplitude (dimensionless)

The ratio of the maximum deformation, measured from the mean deformation, to the free length (thickness) of an unstrained test piece (zero to peak on one side only) in the direction of loading.

3.1.10 root-mean-square strain (dimensionless)

The square root of the mean value of the square of the strain averaged over one cycle of deformation.

NOTE – For a symmetrical sinusoidal strain, the root-mean-square strain equals the strain amplitude divided by $\sqrt{2}$.

3.2 Terms applying to sinusoidal motion

NOTE 1 A sinusoidal response to a sinusoidal motion implies hysteresis loops which are or can be considered to be elliptical. The term "incremental" may be used to designate dynamic response to sinusoidal deformation about various levels of mean stress or mean strain (for example, incremental spring constant, incremental elastic shear modulus).

NOTE 2 For large sinusoidal deformations, the hysteresis loop will deviate from an ellipse since the stress-strain relationship of rubber is non-linear and the response is no longer sinusoidal.

3.2.1 spring constant, *k* (N/m)

The component of applied force which is in phase with the deformation, divided by the deformation.

3.2.2 elastic shear modulus (storage shear modulus), *G′* (Pa)

The component of applied shear stress which is in phase with the shear strain, divided by the strain.

3.2.3 elastic normal modulus (storage normal modulus; elastic Young's modulus), *E′* (Pa)

The component of applied normal stress which is in phase with the normal strain, divided by the strain.

3.2.4 damping constant, *c* (N.s/m)

The component of applied force which is in quadrature with the deformation, divided by the velocity of the deformation.

3.2.5 loss shear modulus, ***G″*** **(Pa)**

The component of applied shear stress which is in quadrature with the shear strain, divided by the strain.

3.2.6 loss normal modulus (loss Young's modulus), *E″* (Pa)

The component of applied normal stress which is in quadrature with the normal strain, divided by the strain.

3.2.7 complex shear modulus, G^* (Pa)

The ratio of the shear stress to the shear strain, where each is a vector which may be represented by a complex number.

$G^* = G' + jG''$

3.2.8 complex normal modulus (complex Young's modulus), E^* (Pa)

The ratio of the normal stress to the normal strain, where each is a vector which may be represented by a complex number.

$E^* = E' + jE''$

3.2.9 absolute (value of) complex shear modulus

The magnitude of the complex shear modulus.

$$\boldsymbol{G}^* = |G^*| = \sqrt{G'^2 + G''^2} \qquad \text{(Pa)}$$

3.2.10 loss factor tanδ (dimensionless)

The ratio of the loss modulus to the elastic modulus. For shear stresses $\tan\delta = G''/G'$ and for normal stresses $\tan\delta = E''/E'$.

3.2.11 loss angle (rad)

The phase angle between the stress and the strain, the tangent of which is the loss factor.

3.3 Other terms applying to periodic motion

3.3.1 logarithmic decrement, Λ (dimensionless)

The natural (Naperian) logarithm of the ratio between successive amplitudes of the same sign of a damped oscillation.

3.3.2 damping ratio, u (dimensionless)

The ratio of the actual to the critical damping, where critical damping is that required for the borderline condition between oscillatory and non-oscillatory behaviour. The damping ratio is a function of the logarithmic decrement:

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

NOTE – $u = \Lambda 2\pi$ for small values of Λ.

3.3.3 dynamic spring rate, K_0

$K_0 = F_0/x_0$

3.3.4 damping coefficient, C

$C = 1/\omega K_0 \sin\delta$

where $\omega = 2\pi f$

3.3.5 transmissibility, V_T

$$V_T = \sqrt{\frac{1+(\tan\delta)^2}{\left[1-\left(\frac{\omega}{\omega_0}\right)^2\right]^2+(\tan\delta)^2}}$$

where ω_0 is the natural angular frequency of the measured undamped vibrator:

$$\varpi_0 = \sqrt{\frac{K^1}{m}} = \sqrt{\frac{K^1 g}{\text{preload}}} \quad \text{and} \quad K^1 = K_0 \cos\delta$$

4 Symbols

A	test piece cross-sectional area
$a(\theta)$	Williams, Landel, Ferry (WLF) shift factor
a	angle of twist
b	test piece width
c_p	heat capacity
γ	strain
γ_0	maximum strain amplitude
δ	loss angle
E	Young's modulus
E_c	effective Young's modulus
E'	elastic modulus
E''	loss normal modulus
E^*	Complex normal modulus (complex Young's modulus)
F	force
f	frequency
G	shear modulus
G'	in phase or storage shear modulus
G''	out-of-phase or loss shear modulus
G^*	complex shear modulus
$\lvert G^* \rvert$	magnitude of complex shear modulus
h	test piece thickness
θ	absolute temperature (in kelvins)
θ_G	low-frequency glass transition temperature
θ_0	reference temperature
k	numerical factor
k_1	shape factor in torsion
l	test piece length
λ	extension ratio
Λ	logarithmic decrement
M'	in-phase or storage modulus
M''	out-of-phase or loss modulus
M^*	complex modulus
$\lvert M^* \rvert$	magnitude of complex modulus
m	mass
p	rubber density
Q	torque
s	shape factor
S'	in-phase component of stiffness
S''	out-of-phase component of stiffness
t	time
$\tan\delta$	tangent of the loss angle

τ stress
τ_0 maximum stress amplitude
τ' in-phase stress
τ'' out-of-phase stress
u damping ratio
ω angular frequency
x displacement

5 Basic principles

5.1 Types of dynamic test

There are two basic classes of dynamic test, i.e. free vibration in which the test piece is set into 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. Forced-vibration test machines may operate at resonance or away from resonance. Wave propagation (e.g. ultrasonics) is a special form of forced vibration and rebound resilience is a simple form of dynamic test in which one half cycle of deformation is applied.

5.2 Dynamic motion

Rubbers are viscoelastic materials and hence their response to dynamic stressing 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

= $_0$sin t (see figure 2) ..(2)

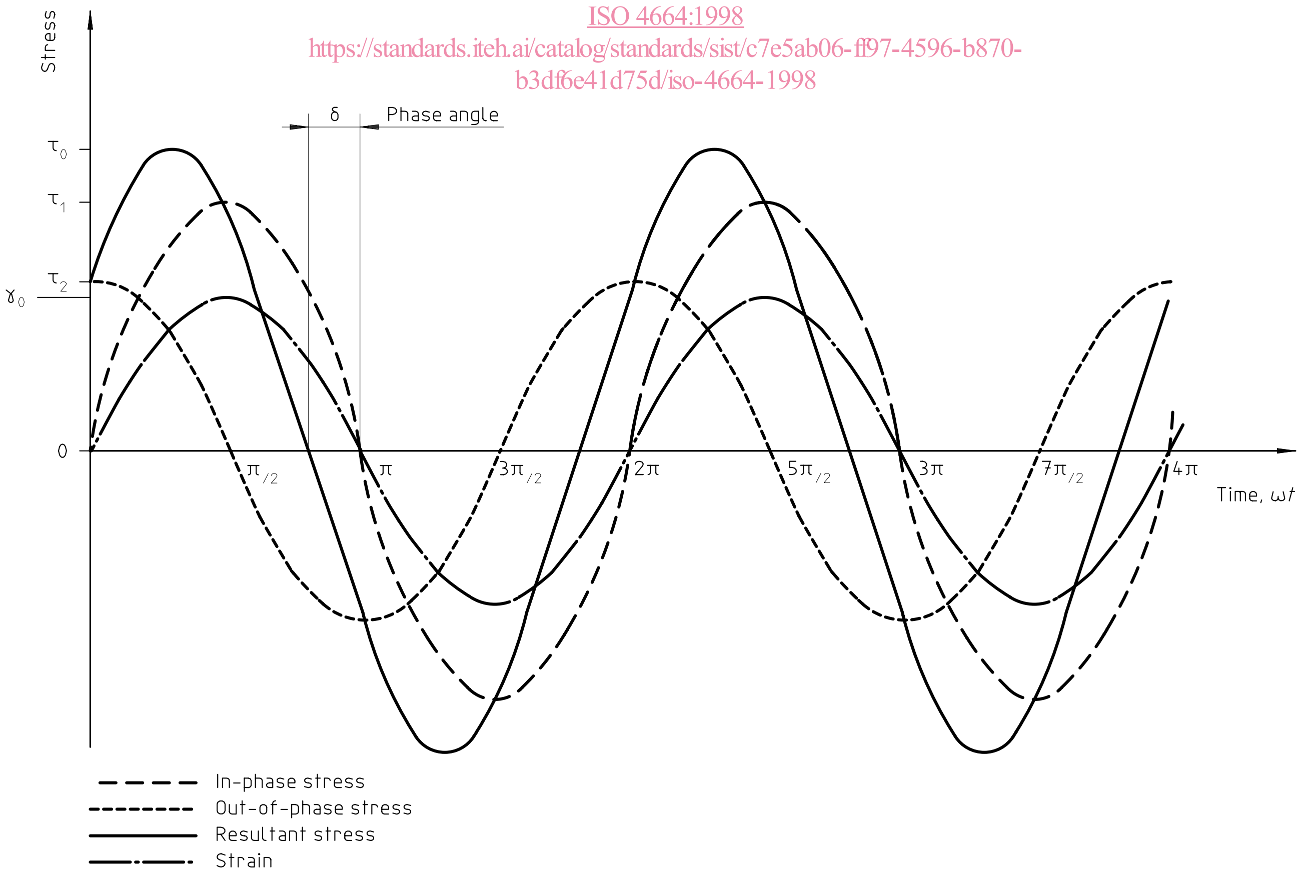


Figure 2 – Sinusoidal stress-strain time cycles

γ γ ω

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

$$\tau = \tau_0 \sin(\omega t + \delta) \qquad ..(3)$$

Considering the stress as a vector having two components, one in phase with the displacement (τ') and one 90° out of phase (τ'') and defining the corresponding in-phase and out-of-phase moduli, the complex (resultant) modulus M^* is given by the following equation:

$$M^* = M' + jM'' \qquad ...(4)$$

Also

$$M' = \frac{\tau'}{\gamma_0} = \frac{\tau_0}{\gamma_0} \cos\delta = M^* \cos\delta$$

$$M'' = \frac{\tau''}{\gamma_0} = \frac{\tau_0}{\gamma_0} \sin\delta = M^* \sin\delta$$

The absolute value, or magnitude, of the complex modulus is given by the following equation:

$$|M^*| = \sqrt{M'^2 + M''^2} \qquad ...(5)$$

The loss tangent, $\tan\delta = \frac{M''}{M'}$

For a freely vibrating rubber and mass system, the equation of motion is given by the following equation:

$$m\frac{d^2x}{dt^2} + \frac{S''dx}{\omega dt} + S'x = 0 \qquad ...(6)$$

The solution of this equation gives

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

$$S'' = \frac{m\omega^2\Lambda}{\pi}$$

$$\tan\delta = \frac{\Lambda}{\pi\left(1 + \frac{\Lambda^2}{4\pi^2}\right)} \qquad ...(7)$$

where Λ is the log decrement.

5.3 Use of dynamic-test data

The reasons for measuring dynamic properties can be given, in general terms, as follows:

a) material characterization
b) design data
c) product performance

Dynamic measurements made as a function of temperature are used to determine the glass transition temperature.

Because of the complex viscoelastic behaviour of elastomers, results of dynamic measurements are highly sensitive to test conditions such as frequency, amplitude of applied force or deformation, test piece geometry and mode of deformation. Hence, such parameters need to be specified and controlled if results are to be comparable.

An important practical consequence is that the conditions under which data is produced should be suitable for the intended purpose of the data. In turn, this may mean that, depending on the intended purpose, a different type of test machine may be suitable. In particular, small dynamic analyser machines which are especially suitable for material characterization may not be capable of operating at the frequencies, amplitudes and modes of deformation required for generating design data or for measuring product performance.

6 Mode of deformation

Dynamic tests are most frequently carried out in shear and compression, but tension and bending are also used. For free-vibration tests, torsion is normally used.

The preferred form of impressed strain is sinusoidal, and the strain should be impressed on the test piece with a harmonic distortion which is as low as possible, and in no case greater than 10 %.

The preferred mode for the generation of design data is simple shear with constant impressed strain. This has the merits that a substantial proportion of manufactured articles are used in this type of strain and the stress-strain behaviour is more nearly linear than in compression or tension, especially for rubbers containing little filler. Forced oscillations rather than free vibration or resonance are preferred because this ensures control of the strain amplitude.

For material characterization, and particularly comparison of materials and quality control, the mode of deformation may be less important than experimental convenience.

For products, the mode of deformation will normally simulate service use.

7 Test pieces

7.1 Test piece preparation

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

7.2 Test piece dimensions

7.2.1 General

Test piece shape and dimensions will vary depending on the mode of deformation and the type and capacity of machine used.

For test pieces bonded to metal plates during moulding, the thickness of the metals should be measured before moulding and the thickness of the rubber deduced by measurement of the overall thickness of the moulding.

7.2.2 Shear

Double shear test pieces are preferred with either round or square rubber elements. It is essential that the diameter (or side in the case of square elements) is at least four times the thickness to ensure that the deformation is essentially simple shear, i.e. bending is negligible. The thickness should be no greater than 12 mm to avoid difficulties in obtaining uniform vulcanization.

The thickness and area of each test piece should be measured to ± 1 %.

7.2.3 Compression

Cylindrical test pieces are preferred with a height/diameter ratio of approximately 1,5. This ratio minimizes uncertainties due to shape factor correction for unlubricated test pieces. However, the test pieces specified in ISO 815 are convenient and widely used.

The thickness and area of each test piece should be measured to ± 1 %.

7.2.4 Tension

Rectangular test pieces are preferred of thickness between 1 mm and 3 mm and length between the grips five times the width.

The thickness, width and gauge length should be measured to ± 1 %.

7.2.5 Bending

Rectangular test pieces are preferred of thickness between 1 mm and 3 mm. For three- or four-point loading, the span should ideally be 16 mm, but this may have to be reduced to obtain an adequate measurable force. In this case, the maximum practicable value should be used, and it should be accepted that the deformation will have a significant shear component.

Measure the span, width and thickness to ± 1 %.

7.2.6 Torsion

Rectangular test pieces are preferred of thickness between 1 mm and 3 mm, width between 4 mm and 12 mm (subject to a maximum width to thickness ratio of 10) and length between the grips at least 10 times the width (subject to a maximum of 120 mm).

The thickness, width and distance between the grips should be measured to ± 1 %.

7.2.7 Conical shear

Circular test pieces of about 35 mm diameter are suitable. For cone/cone geometry a cone angle of less than 5°, and for cone/plate geometry a cone angle of less than 3°, is required to maintain uniform shear.

The test pieces may be cured *in situ* and tested with a minimum compressive force of 11,5 kN to avoid the need for adhesive bonding.

7.3 Products

Test pieces of dimensions given in 7.2 may be obtained from some products by cutting and buffing. In other cases, it may be necessary or desired to test the complete product.

7.4 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 are tested.

8 Test apparatus

8.1 Classification

There is a great variety of dynamic test apparatus in use and several ways in which it can be classified. The basic classification is between free and forced vibration, with the latter type operating either at resonance or away from resonance.

Apparatus can also be classified on the basis of the mode of deformation, and for forced vibration the type of drive and whether the load or deformation amplitude is held constant during the test.