International Standard 4662

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Rubber — Determination of rebound resilience of vulcanizates

Caoutchouc — Détermination de la résilience de rebondissement des vulcanisats

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0 Introduction

When rubber is deformed, an energy input is involved, part of which is returned when the rubber returns to its original shape. That part of the energy which is not returned as mechanical energy is dissipated as heat in the rubber.

The ratio of the energy returned to the energy applied is termed the resilience. When the deformation is an indentation due to a single impact, this ratio is termed the rebound resilience.

The value of the rebound resilience for a given material is not a fixed quantity, but varies with temperature, strain distribution (determined by the type and dimensions of the indentor and test piece), strain rate (determined by the velocity of the indentor,), strain energy (determined by the mass and velocity of the indentor) and strain history. Strain history is particularly important in the case of filler-loaded polymers, where the stress-softening effect necessitates also a mechanical conditioning.

This variation of resilience with conditions is an inherent property of polymers, which can therefore only be fully evaluated if tests are carried out over a wide range of conditions. The factors described may have a different quantitative influence on resilience; while temperature may critically affect resilience near transition regions of the material tested, factors connected with time and amplitude of indentation have moderate effects and fairly wide tolerances may be admissible for them.

Ideally, rebound resilience should be measured on a test piece the back surface of which is bonded to a rigid support in order to avoid friction losses due to slippage during the impact. Since the use of bonded test pieces is impractical in many applications, unbonded test pieces are used. These require secure clamping to avoid frictional movements.

To approach these ideal conditions in a practical apparatus it is necessary to put limitations upon the hardness (see ISO 48) of the rubber that may be tested: on the hard side to avoid unusual requirements of rigidity in the apparatus; on the soft side to avoid difficulties in clamping.

If a defined set of mechanical conditions and an appropriate apparatus are selected, a standard value of rebound resilience at any temperature can be obtained with a satisfactory degree of reproducibility.

1 Scope and field of application

This International Standard specifies a method of determining the rebound resilience of rubber in one narrow range of impact strain and strain rate, by means of any form of impacting and measuring apparatus conforming to the requirements described below.

The test method is applicable to vulcanized rubbers, the hardness of which, at the test temperature, lies between 30 and 85 IRHD.

2 References

ISO 48, *Vulcanized rubber — Determination of hardness (Hardness between 30 and 85 IRHD).*

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1826, *Rubber, vulcanized — Time-interval between vulcanization and testing — Specification.*

ISO 2856, *Elastomers — General requirements for dynamic testing.*

ISO 3383, *Rubber — General directions for achieving elevated or sub-normal temperatures for tests.*

ISO 4661/1, *Rubber, vulcanized — Preparation of samples and test pieces — Part 1: Physical tests.*

3 Definition

The terminology used in this International Standard, as well as the general testing conditions, are in agreement with ISO 2856, to which reference is also made for the theory behind the test. In addition the following term is used:

standard rebound resilience: The ratio between the returned and applied impact energy when a spherically terminated mass impacts a flat test piece, firmly held while free to bulge, the impacting mass and indentor and impacted test piece characteristics being such as to fall within the following specified ranges:

indentor diameter (D): 12,45 to 15,05 mm test piece thickness (d) : 12,5 ± 0,5 mm

impacting mass (m) : 0,35 $_{-0,1}^{\ 0}$ kg

impact velocity (v) : 1,4 $^{+0,6}_{\ 0}$ m/s

apparent strain energy density (mv^2/Dd^2) :

351 $^{+112}_{-27}$ kJ/m³

NOTES

1 The conditions and apparatus specified in this International Standard therefore involve the selection of a spherical indentor and of a flat piece and are assumed to be essentialy dependent on the fundamental magnitudes D, d, m and v listed above. In addition, the ratio of impact energy to an equivalent volume, or "apparent strain energy density" (mv^2/Dd^2), which under simplifying assumptions is related to impact strain, has to be maintained whithin the narrow range specified.

2 The stated nominal characteristics (12,5 mm; 12,5 mm; 0,35 kg; 1,4 m/s; 351 kJ/m³) are the same as those prescribed for the Lüpke pendulum method. The tolerances are such as to include the nominal characteristics for the modified Schob pendulum method (15,0 mm; 12,5 mm; 0,25 kg; 2 m/s; 427 kJ/m³).

In addition allowance has been made for :

— a small tolerance (± 0,05 mm) to allow for mechanical imperfections of spheres of 12,5 and 15 mm nominal diameter;

— an additional tolerance ($^{+36}_{-27}$ kJ/m³) on mv^2/Dd^2, to allow for the effect of variation of test piece thickness (± 0,5 mm).

4 Apparatus

4.1 General

The rebound resilience shall be measured by means of an apparatus consisting of a pendulum-like one-degree-of-freedom mechanical oscillatory device and a heavy and secure test piece holder.

The two items shall be suitably fixed together for rebound resilience measurements, and either may be removed for purposes of adjustment or checking of the oscillatory device. Means shall be provided for measuring the rebound of the pendulum, either on a calibrated scale or as an electrical signal.

Various practical designs of apparatus, which conform to these specifications, are available (see annex B).

NOTE — Types of apparatus designed to operate within the ranges specified for the various parameters and correctly calibrated give substantially the same values of rebound resilience.

4.2 Oscillatory device

The oscillatory device shall consist of a rigid body or hammer terminated by an indenting spherical surface, supported so as to oscillate linearly or circularly under the action of a restoring force, which may be due to gravity or elastic reactions of springs, or to a wire in torsion. A system of pointer and fixed scale shall be provided to follow the motion of the hammer.

The velocity of the indenting spherical surface at the point of impact shall be in the horizontal direction.

4.2.1 Scale (see figure 1)

For pendulums in which the restoring force is due to gravity, the rebound resilience R is given by

$$R = \frac{h}{H}$$

where

h is the height of rebound;

H is the height of fall.

It is usually convenient for the scale to measure either the horizontal rebound distance or, for rigid arm pendulums in particular, the angle of rebound. For pendulums on which the restoring force is due to a torsion wire or elastic reactions of springs, the rebound resilience is given by

$$R = \frac{\alpha_R}{\alpha_I}$$

where

α_R is the angle of rebound;

α_I is the angle of impact.

For this form of apparatus it is convenient for the scale to measure the angle of rebound.

The scale may be graduated uniformly or be calibrated directly in units of resilience. For uniformly graduated scales, conversion equations, charts or tables to allow the determination of resilience are also necessary.

4.2.2 Adjustment of oscillatory device

The complete apparatus shall be repeatedly operated, impacting pieces of rubber at the extreme ranges of hardness; its motion shall be smooth and no form of spurious oscillation mode, such as whip or vibration, shall be caused by the impact because of insufficient stiffness of rigid parts or a defective system of guidance.

For the purpose of initial adjustment or periodic checking, the test piece holder shall be removed from the oscillatory device and the following procedure carried out.

4.2.2.1 Direct weighing and geometric measurements of the moving hammer and of its distances from the guiding pivots or suspensions shall be made in order to carry out inertial parameter calculations. From these it shall be determined that the equivalent impacting mass conforms to the specifications in clause 3 and that its line of impact is such as not to cause significant reactions on pivots or suspensions.

Ensure that the diameter of the indenting spherical surface conforms to the specifications in clause 3 and that the spherical surface of the indentor in all cases exceeds the indented surface of the rubber; it is preferable that the indentor be a complete half-sphere.

4.2.2.2 The complete oscillatory device shall be left free to attain its rest position. Check that this is on the zero point of the scale, which shall also be the position at which impact takes place; at this point the indenting sphere shall be moving horizontally.

4.2.2.3 To correct for frictional losses, the oscillatory device shall be set in motion; its period of oscillation shall be timed, and the decrease of successive amplitudes, on the same side, followed and noted. The corresponding logarithmic decrement Λ shall be calculated from the expression

$$\Lambda = \frac{1}{n} \log_e \frac{l_x}{l_{x+n}}$$

$$= \frac{1}{2n} \log_e \frac{R_x}{R_{x+n}}$$

where

n is the number of full oscillations considered;

l_x and l_{x+n} are amplitudes read on a uniform scale;

R_x and R_{x+n} are amplitudes read on a quadratic scale.

For the present purpose it is immaterial whether the scale has or has not already been corrected for small non-linearities (see 4.2.2.5).

If the operation of the instrument involves different damping conditions during the forward and backward impact strokes, due for example to a pawl engaging the pointer, then the described measurements shall be carried out under both conditions and their readings averaged.

4.2.2.4 Full period (T) and logarithmic decrement (Λ) shall be calculated as averages of five oscillations for different amplitudes as follows:

full scale	T_1	Λ_1
one-half scale	T_2	Λ_2
one-quarter scale	T_4	Λ_4

4.2.2.5 None of the values T_1, T_2 and T_4 shall differ from their average by more than 10 %. While a difference under 1 % may be neglected, a difference between 1 % and 10 % shall be taken into account through suitable non-linearity corrections in the scale. These shall be made with reference to the pendulum energy corresponding to the scale point.

The value of impact velocity shall be checked from geometric dimensions and the average of T_1, T_2 and T_4 or from values of mass and energy at unity resilience point. It must conform to the specifications in clause 3.

4.2.2.6 None of the values Λ_1, Λ_2 and Λ_4 shall differ from their average be more than 0,01 and none of them shall exceed 0,03. While a value under 0,01 may be neglected, for values between 0,01 and 0,03 a correction to the rebound results shall be provided. This correction shall have the value of Λ, and shall be applied to the results preferably by displacing the starting point of the moving mass beyond the unity resilience point by a corresponding amount.

A more refined evaluation of the correction is in most cases unnecessary, but can be made if a detailed analysis of energy losses in available.

4.3 Test piece holder

4.3.1 The disc test piece shall be securely held during mechanical conditioning and rebound measurement.

The surface against which the back of the test piece is applied shall be metallic, flat and smoothly finished, vertical and perpendicular to the impact velocity direction.

This flat face is a part of an anvil which shall, if free, have a mass of at least 200 times the impacting mass, or shall be rigidly fastened to a very rigid system, such as a masonry structure.

Any type of suitable holding device may be used provided that it gives rebound resilience values that deviate not more than 0,02 (absolute rebound resilience) from those obtained with test pieces bonded to a rigid back plate. This shall be checked using one compound of high rebound resilience (approximately 0,90) and one of high hardness (approximately 85 IRHD).

Examples of suitable holding devices include suction holding (by vacuum), mechanical clamping devices and combinations of the two. A recommended mechanical clamping device consists of a metal ring (see figure 2); this shall have 20 mm internal diameter and 35 mm external diameter and shall exert on the front of the test piece a force of 200 ± 20 N given, for example, by springs. The indenting sphere shall enter, at its rest position, the centre of the retaining ring. Another recommended method of holding is by applying suction on the back of the test piece; this should be done though a circular groove 25 mm diameter and 2 mm width, by a pump which shall maintain an absolute pressure not greater than 10 kPa; in this case, the force exerted by the retaining ring may be reduced to 150 ± 15 N.

No lateral restraint shall be applied to the test piece; a clearance of at least 2 mm shall be left around it in order to allow it to bulge freely when impacted.

4.3.2 If measurements are to be carried out at a series of temperatures different from the ambient temperature, the pendulum may be placed and operated in a suitable oven or cold chamber operating in accordance with ISO 3383. In this case the apparatus shall be checked for correct operation (see 4.2.2) in the range of temperatures used. Alternatively, suitable provisions shall be made for heating or cooling the test piece holder by means of circulating fluids (see figure 3). A heated or cooled gas curtain on the front opening of the holder is recommended in order to ensure that the test piece is completely surrounded by a temperature-controlled medium.

Thermocouples or other means shall be provided for measuring the temperature of the hodler in a position as close as possible to the test piece.

5 Test piece

5.1 Preparation

The test pieces shall be prepared in accordance with ISO 4661/1, either by moulding or be cutting. They shall be free of fabric or any other reinforcing support.

The test pieces shall have flat, smooth and parallel surfaces, finished, if necessary, by buffing. If stickness is shown on the impacted surface, its effect shall be avoided by dusting the surface lightly, for example with talc.

5.2 Dimensions

The standard test piece shall be a disc with thickness 12,5 ± 0,5 mm and dimeter 29 ± 0,5 mm Other test pieces having non-standard dimensions may be used for comparative measurements with special provisions (see annex A).

5.3 Measurement of dimensions

The thickness of the test piece shall be measured with an accuracy of 0,05 mm and its diameter with an accuracy of 0,2 mm.

5.4 Number of test pieces

For each material, two test pieces shall be tested in succession.

5.5 Conditioning

5.5.1 The time-lapse between vulcanization and testing shall be in accordance with ISO 1826.

5.2.2 Samples and test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

5.5.3 If the test piece is buffed, the interval between buffing and testing shall not exceed 72 h.

5.5.4 Prepared test pieces shall be conditioned, immediately before testing, at a standard laboratory temperature complying with ISO 471.

6 Temperature of test

The standard laboratory temperature (see 5.5.4) is the preferred temperature of test. Tests may also be carried out at one or more of the following temperatures: −75, −55, −40, −25, −10, 0, 40, 55, 70, 85, 100 °C. Tolerance on temperature shall be not more than ±1 °C.

Where resilience changes quickly with temperature, other temperatures at smaller intervals may be used.

7 Procedure

7.1 Thermal conditioning

If the test temperature differs from the standard laboratory temperature (see 5.5.4), first bring the complete testing apparatus, or the special heated or cooled holder (see 4.3.2), to the test temperature.

Mount the test piece in the holder and allow sufficient time for the test piece to reach a uniform temperature within the prescribed tolerance (see ISO 3383). Alternatively, test pieces may be heated or cooled separately from the holder in an oven or cold chamber in accordance with ISO 3383 and then quickly inserted in the heated or cooled holder; in this case the time in the holder before testing may be reduced to 3 min.

In tests at low temperatures, provision shall be made to prevent frost from forming on the test piece.

7.2 Mechanical conditioning of test piece

After applying the prescribed thermal conditioning and mounting the test piece in the appropriate holding device, carry out a mechanical conditioning by subjecting the test piece to not fewer than the three and not more than seven successive impacts, so as to reach a practically constant rebound amplitude.

7.3 Measurement of rebound resilience

Immediately after the impacts for mechanical conditioning, apply three more impacts at the same velocity to the test piece and note the three rebound readings.

Convert these three readings, if necessary (see 4.2.1), to resilience values, expressed as a percentage: their median shall be taken as the rebound resilience of the test piece.

Calculate the average of the values for the two test pieces.

8 Test report

The test report shall include the following particulars:

a) Sample details:

1) full description of the sample and its origin;

2) compound details and curing conditions, if known;

3) preparation of test pieces, for example whether moulded or cut;

4) any relevant facts about the pre-test history of the test pieces.

b) Test method and test details:

1) reference number of this International Standard and corresponding national standard (if available);

2) type of apparatus used, and value of diameter of indentor, its mass and velocity;

3) method of holding test piece;

4) test temperature;

5) time and temperature of conditioning of test pieces prior to testing;

6) any non-standardized procedures adopted.

c) Test results:

1) number of test pieces tested;

2) calculated mean value of rebound resilience, in per cent, for the test pieces tested, according to 7.3.

d) Data of test.

Annex A

Non-standard test pieces

(This annex does not form part of the Standard.)

A.1 Non-standard diameter

Test pieces of standard thickness but having a diameter larger than standard, up to a maximum of 53 mm, may be tested, giving results close to standard rebound resilience with the standard procedure and apparatus, provided that an increased holding force is applied (see table).

A.2 Non-standard thickness

Resilience of test pieces having a thickness differing from, and in general lower than, 12,5 ± 0,5 mm, may be measured to give results close to the standard rebound resilience, using two different methods, both based on the principle of maintaining the same constant value of apparent strain energy density mv^2/Dd^2 = 351 kJ/m^3.

A.2.1 Method A: impact velocity adjustment

The impact velocity v is changed proportionnaly to the test piece thickness. This reduction of velocity may be easily obtained by changing the starting point and initial deviation of the impacting hammer, whilst keeping the indentor diameter and impacting mass at the standard values (see clause 3).

In order to hold thinner test pieces while maintaining the same internal diameter of the holding ring, the latter shall be supplemented by the use of suction (see 4.3.1).

While this method gives results which may show some discrepancies from standard rebound resilience, because of different strain distribution and poorer test piece holding, it has the advantage of being cheaper because only one apparatus, with minor adjustments, may accommodate different thicknesses.

A.2.2 Method B: mechanical similitude

The indentor diameter D, the impacting mass m and the impact velocity v are all changed proportionally to test piece thickness (see the table). At the same time the test piece diameter and holding ring and force have to be altered to suit the changed thickness (see the table and figure 4).

This method obviously requires various sizes of apparatus for its application and, unless only a single non-standard thickness is to be tested, is therefore more expensive; it has the important advantage of giving results very close to the standard rebound resilience.

A.2.3 Stacked test pieces

A stack, of no more than three thin test pieces of the same material, may be used to obtain a greater thickness. It is necessary that the surfaces of the test pieces be very smooth, and lateral suction may help in ensuring their contact.

Stacking of test pieces introduces additional uncertainties, and shall be used only for comparative measurements.

A.3 Thermal conditioning of non-standard test pieces

The thermal conditioning of non-standard test pieces may be carried out as for the standard ones (see 7.1), but taking into account the changed dimensions.

Roughly, the time, in minutes, allowed for reaching uniform temperature should be not less than 1,5 times the thickness, in millimetres. In no case shall this time be less than 3 min.

A.4 Test report

In addition to the data already required (see clause 8), the dimensions of the test piece and the method and apparatus used to accommodate non-standard dimensions shall be reported.

Table — Recommended fundamental parameters, based on the mechanical similitude principle (method B), to be used when handling test pieces of non-standard dimensions

	Size I	Size II	Size III	Size IV	Size V
Fundamental characteristics					
Test piece thickness d mm	2 ± 0,1	4 ± 0,2	6,3 ± 0,3	12,5 ± 0,5	25 ± 1
Indenting sphere diameter D mm	2 ± 0,05	4 ± 0,1	6,3 ± 0,1	12,5 ± 0,1	25 ± 0,2
Inpacting mass m kg	0,056 ± 0,001	0,112 ± 0,002	0,176 ± 0,005	0,35 ± 0,01	0,70 ± 0,01
Impacting velocity v m/s	0,222 ± 0,005	0,45 ± 0,005	0,71 ± 0,01	1,40 ± 0,01	2,80 ± 0,02
Recommended mechanical clamping					
Test piece diameter					
minimum mm	9	15	20	29	50
maximum mm	25	45	53	53	70
Retaining ring diameter (see figure 4)					
inner mm	5	8	12	20	36
outer mm	10	16	22	35	55
Force on test piece N	50	100	150	300	600

NOTE — The holding force of 300 N for size IV refers to a maximum diameter of 53 mm, while the force of 200 N appearing in 4.3.1 refers to a maximum diameter of 35 mm. The holding force necessary to obtain consistent values of resilience is contained within a widely varying range depending on vulcanizate dimensions and properties, finish of holding surfaces and previous contact history. Its maximum value is limited by possible excessive deformation of soft vulcanizates, while its minimum value is limited by possible shuffle or slip.

The values here indicated are considered to be safe values for most ranges of dimensions and properties involved.