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Rubber, vulcanized — Determination of tension fatigue

Caoutchouc vulcanisé — Détermination de la fatigue en traction

Reference number
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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.

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

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

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

ISO 6943 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 6943:2007), which has been revised primarily to include a calibration schedule for the apparatus used (see Annex B).

Rubber, vulcanized — Determination of tension fatigue

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.

IMPORTANT — Certain procedures specified in this International Standard might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This International Standard describes a method for the determination of the resistance of vulcanized rubbers to fatigue under repeated tensile deformations, the test piece size and frequency of cycling being such that there is little or no temperature rise. Under these conditions, failure results from the growth of a crack that ultimately severs the test piece.

The method is restricted to repeated deformations in which the test piece is relaxed to zero strain for part of each cycle. Analogous fatigue processes can occur under repeated deformations which do not pass through zero strain and also, in certain rubbers, under static deformation, but this International Standard does not apply to these conditions.

The method is believed to be suitable for rubbers that have reasonably stable stress-strain properties, at least after a period of cycling, and that do not show undue stress softening or set, or highly viscous behaviour. Materials that do not meet these criteria might present considerable difficulties from the points of view of both experiment and interpretation. For example, for a rubber that develops a large amount of set during the fatigue test, the test strain will be ill-defined and the fatigue life is likely to differ markedly under constant maximum load and constant maximum extension conditions; how the results for such a rubber should be interpreted, or compared with those for other rubbers, has not been established by basic work. As a general guide, a rubber for which the set determined in accordance with 9.5 and 10.2 exceeds 10 % is likely to fall into this category. For this reason, the method is not considered suitable for most thermoplastic elastomers.

Similar considerations apply with regard to other changes in elasticity behaviour during testing.

A distinction should be made between this fatigue test and the flexometer tests described in the various parts of ISO 4666, where fatigue breakdown occurs under the simultaneous action of stress and temperature.

Advantages over the De Mattia flex cracking and cut growth test (see ISO 132) include the following. The test yields quantitative results which do not depend on operator interpretation and which can be recorded automatically. The initial deformation is clearly defined and can readily be varied to suit different applications.

Great caution is necessary in attempting to relate standard test results to service performance since the comparative fatigue resistance of different vulcanizates can vary according to the test conditions used and to the basis by which the results are compared. Guidance on the selection of test conditions and on the interpretation of results is given in Annex A.

2 Normative references

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

ISO 18899:2004, *Rubber — Guide to the calibration of test equipment*

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.

3.1
fatigue life
number of cycles required to break a test piece repeatedly deformed to a prescribed tensile strain

3.2
tension fatigue
fracture, through crack growth, of a component or test piece subjected to a repeated tensile deformation

4 Principle

Dumb-bell or ring test pieces are repeatedly deformed in simple extension until they fail by breaking. The test pieces are relaxed to zero strain for part of each cycle. The number of deformation cycles to failure, defined as the fatigue life, is determined as a function of the maximum strain and, if required, as a function of the maximum stress or strain energy density imposed during the test.

5 Apparatus

5.1 Fatigue-testing machine

The fatigue-testing machine shall provide a reciprocating motion at a frequency which shall normally be within the range 1 Hz to 5 Hz.

For testing dumb-bell test pieces, the machine shall be provided with clamps that grip the test piece sufficiently firmly to prevent slippage, irrespective of the magnitude of the strain applied.

For testing ring test pieces, each station on the machine shall be provided with two pairs of rollers, one pair fixed to the body of the machine and the other to the reciprocating part. To minimize friction, the rollers shall be fabricated from stainless or chromium-plated steel, well polished and fitted with free-running ball races. The roller arrangement shall be such that the test pieces are held securely in place over the rollers throughout the test.

The stroke of the machine and the position of the fixed clamps or rollers shall be adjustable to provide a range of test strains. In all cases, the test piece shall be relaxed to zero strain for part of each cycle.

The fixed clamps or rollers should preferably be fitted with contacts or other means of operating counters to register the number of cycles to failure of each test piece.

If it is required to determine the maximum stress of the cycle, manual or automatic means for measurement of the load shall be provided. Stress-strain properties and strain energy density under test conditions can be determined for rings if automatic equipment for force-extension measurement is provided.

Alternatively, and for dumb-bell test pieces, stress-strain properties can be determined separately using a conventional tensile-testing machine.

5.2 Dies and cutters

All dies and cutters used shall be made and maintained in accordance with ISO 23529.

Since fatigue life is sensitive to flaw size, it is essential that the dies or cutters used for the preparation of test pieces be carefully maintained so that the cutting edges are sharp and free from nicks. Regular control tests, using an established rubber, shall be made to check sharpness. Any oil shall be removed from the cutter after sharpening.

5.3 Marker

If a marker is used for marking the reference lines on dumb-bell test pieces, it shall have two parallel edges. These shall be ground smooth and true, 0,05 mm to 0,10 mm wide at the edge and bevelled at an angle of not more than 15°.

The marking implement shall not damage the rubber surface.

5.4 Marking substance

The marking substance shall have no deleterious effect on rubber and shall be of contrasting colour.

5.5 Measuring instruments

The instrument for measuring the thickness of dumb-bell test pieces (and the axial thickness of ring test pieces) shall be in accordance with ISO 23529, consisting essentially of a micrometer dial gauge having a circular foot which does not extend beyond the surface of the rubber where the measurement is being taken, and applying a pressure of (22 ± 5) kPa for a rubber with hardness equal to or higher than 35 IRHD.

Vernier calipers, a travelling microscope or other suitable means shall be provided for the measurement of other test piece dimensions. A graduated cone is recommended for the measurement of the internal diameter and internal circumference of ring test pieces.

6 Calibration

The requirements for calibration of the test apparatus are given in Annex B.

7 Test piece

7.1 Dimensions

7.1.1 General

Standard test pieces shall be dumb-bells or rings having dimensions within the limits prescribed in 7.1.2 and 7.1.3. Any test piece showing irregularities or imperfections shall not be used.

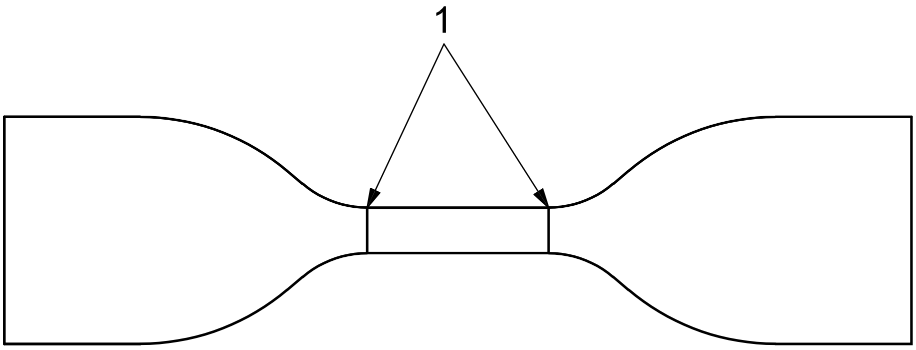
7.1.2 Dumb-bell test piece

Dumb-bell test pieces and the dies with which they are cut out shall be as shown in Figure 1. The dies shall have the dimensions given in Table 1. The reference length (the distance between the marked reference lines) shall be 25 mm for the type 1 test piece and 20 mm for the type 1A and type 2 test pieces. This length shall be equidistant from the ends of the central parallel-sided part of the test piece. The tabs may have beaded ends for location purposes.

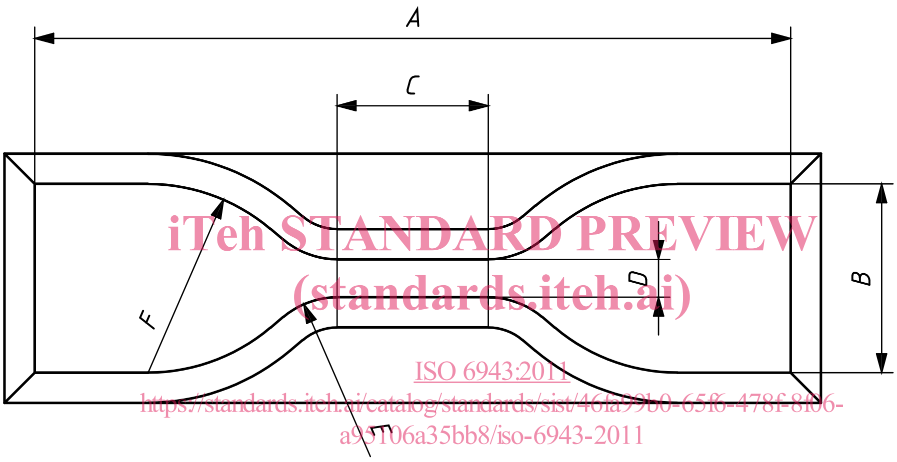
The thickness of dumb-bells shall be (1,5 ± 0,2) mm. In any one dumb-bell, the thickness of the narrow part shall nowhere deviate by more than 2 % from the mean. If results from two sets of dumb-bells are being compared, the mean thicknesses of the sets shall be within 10 % of one another.

Fatigue life depends on test piece thickness and it has been shown that, at a thickness of 1,5 mm, the life is least sensitive to change in this dimension. If required, an alternative thickness of (2,0 ± 0,2) mm may be used provided it is recorded in the test report, but it might lead to different results.

Dumb-bells shall be cut from sheet by punching with a die using a single stroke of a press. The rubber shall be supported on a sheet of slightly yielding material (for example cardboard or polyethylene) on a flat rigid surface; the region of the supporting sheet beneath the die shall be free from cuts or other imperfections. Care shall be taken to ensure that the rubber is isotropic and free from built-in stresses (failure to meet either of these requirements can cause very marked variations in fatigue life); in cases where there is any doubt, check stress-strain and fatigue tests shall be carried out using test pieces cut in different directions or from different locations in a sheet. Any sheet showing such imperfections shall be discarded unless anisotropy or “grain” effects are being investigated, when their extent and direction shall be specified and recorded in the test report.



a) **Test piece**



b) **Die**

Key

1 reference lines

A to *F* see Table 1

Figure 1 — Shape of dumb-bell test pieces and die

Table 1 — Die dimensions for dumb-bell test pieces [see Figure 1 b)]

Dimensions in millimetres

Dimension	Type 1	Type 1A	Type 2
A Overall length, min.	115	100	75
B Width of ends	25 ± 1	25 ± 1	12,5 ± 1
C Length of narrow parallel-sided portion	33 ± 2	21 ± 1	25 ± 1
D Width of narrow parallel-sided portion[a]	6,2 ± 0,2	5 ± 0,1	4 ± 0,1
E Small radius	14 ± 1	11 ± 1	8 ± 0,5
F Large radius	25 ± 2	25 ± 2	12,5 ± 1
[a] The variation within any one die shall not exceed 0,05 mm.			

NOTE The dies are identical to those specified for type 1, type 1A and type 2 dumb-bell test pieces in ISO 37 for the determination of tensile stress-strain properties.

7.1.3 Ring test piece

The standard ring test piece shall have a nominal internal diameter of 44,6 mm and an external diameter of 52,6 mm, giving a nominal radial width of 4 mm; the radial width shall nowhere deviate from the mean by more than 0,2 mm. The axial thickness shall be (1,5 ± 0,2) mm and on any one ring the thickness shall deviate from the mean by no more than 2 %.

NOTE With respect to the internal and external diameters and the tolerance on radial width (but not the axial thickness), the standard ring test piece is identical to the normal-size (type A) ring test piece specified in ISO 37.

Alternative axial thicknesses and radial widths may be used, provided that they are recorded in the test report. These alternatives include an axial thickness of (2,0 ± 0,2) mm and the use of a ring of (2,0 ± 0,2) mm radial width and (3,0 ± 0,2) mm axial thickness, the latter being cut from 3-mm-thick sheet, or from 6-mm-thick sheet and then divided into two. Note that a change in dimensions can change the stress distribution within the cross-section of the deformed test piece and might therefore lead to different results. Comparisons shall only be made between test pieces having the same dimensions.

Rings shall be produced from a sheet by either die-stamping or cutting with revolving knives; in the latter case, water may be used as a lubricant but contact shall be minimized and the rubber allowed to dry thoroughly prior to testing. A substrate shall be used, as for dumb-bells, and similar care shall be taken to ensure that the sheet is isotropic and homogeneous.

7.2 Number of test pieces

The number of test pieces required for the determination of fatigue life at each test strain depends on the purpose of the test and on the inherent variability of the materials being examined. At least five test pieces shall be tested in the case of routine quality control measurements on materials that are already well characterized. For other purposes, and particularly for rubbers that show large variability, more test pieces might be required to obtain a representative result (see 10.1).

Additional test pieces might be required for the determination of stress, strain energy density, and set developed during cycling.

7.3 Storage and conditioning

For all test purposes, the minimum time between vulcanization and testing shall be 16 h, in accordance with ISO 23529; the maximum time shall be 4 weeks unless special circumstances (such as investigation of ageing effects) dictate otherwise.

Test sheets and test pieces shall be stored in the dark at a standard laboratory temperature (see ISO 23529). They shall not, at any time, be allowed to come into contact with test sheets or test pieces of a different composition. This is necessary in order to prevent additives that might affect fatigue life, such as antioxidants, from migrating from one vulcanizate into adjacent vulcanizates.

For tests at a standard laboratory temperature, test pieces shall be conditioned at this temperature for a minimum of 3 h (in accordance with ISO 23529) immediately before testing. For tests at other temperatures, test pieces shall be conditioned at the test temperature immediately before testing for a sufficient period to reach temperature equilibrium.

For tests intended to be comparable, the duration and temperature of storage and the duration and temperature of conditioning shall be the same.

8 Test conditions

8.1 Test strains

The choice and number of test strains will depend on the particular project or application. For test pieces relaxed to zero strain, the test strain is the initial maximum strain imposed during cycling, and for many purposes it will be in the range 50 % to 125 % elongation. Lower or higher strains may be used.

It is strongly recommended that tests be conducted at several test strains so that the dependence of fatigue life on strain, and, if required, on the maximum stress or maximum strain energy density imposed during cycling, can be determined. For this purpose, at least four test strains should be used. The strain intervals required will depend on the range covered and the rate at which the fatigue life varies with strain within that range; as a general guide, intervals of 25 % are suggested, but narrower or wider intervals may be used. It is recommended that the test at the highest maximum strain be carried out first and then the test strain progressively lowered.

The test piece shall return to zero strain for part of each cycle.

8.2 Test frequency

The frequency of cycling shall normally be in the range 1 Hz to 5 Hz, but other frequencies may be used for particular purposes.

For tests intended to be comparable, the frequency shall be the same.

NOTE It has been found that fatigue life is not markedly affected by frequency over the range 1 Hz to 5 Hz, provided that the conditions described in Clause 1 are respected.

8.3 Test temperature

Tests shall normally be carried out at a standard laboratory temperature. Other temperatures may be used if appropriate for particular applications, and these should be selected from the list given in ISO 23529.

NOTE Caution is required in the use of extreme temperatures. For example, at high temperatures, set developed during cycling can be very extensive and can markedly influence the results. At low temperatures, viscosity phenomena can appear if the test temperature approaches the glass-transition temperature, T_g.

8.4 Test atmosphere

The test shall not normally be made in a room which contains any apparatus that generates ozone, such as a fluorescent lamp, or which for any other reason has an ozone content above that in normal indoor air. The motor used to drive the test machine shall be of a type that does not generate ozone.

NOTE Periodic checks are advised in order to ensure the ambient ozone concentration is preferably less than 1 part by volume per 100 million parts of air. When these conditions are observed, the fatigue life should not be significantly affected by the ozone concentration except at strains near to or below the mechanical fatigue limit of the material under test (see Annex A).

9 Procedure

9.1 Marking of dumb-bell test pieces

Mark each test piece with reference lines, using a marker which satisfies the conditions described in 5.3 and 5.4. The test piece shall be marked in the unstrained state and shall not have been strained prior to marking. The reference lines shall not exceed 0,5 mm in width and shall be marked on the narrow part of the test piece at right angles to its edge and equidistant from its centre.

9.2 Measurement of test pieces

9.2.1 Dumb-bell test pieces

Measure the thickness of each test piece at its centre and at each end of the reference length using the thickness gauge described in 5.5. The width of the test piece shall be assumed to be equal to the width between the cutting edges of the narrow, central part of the die. For this purpose, the width of this part of the die shall be measured to the nearest 0,05 mm. The mean value of each set of measurements shall be used in calculating the area of the cross-section.

Using Vernier calipers or other means, measure the distance between the centres of the reference lines to the nearest 0,2 mm. The test piece shall be in the unstrained state and shall not have been strained prior to measurement.

9.2.2 Ring test pieces

Measure the radial width and axial thickness at six positions approximately equally spaced around the circumference of the ring, using the instruments described in 5.5. The mean value of each set of measurements shall be used in calculating the area of the cross-section.

Measure the internal diameter to the nearest 0,2 mm, preferably by means of a suitable cone. The initial unstrained internal circumference, l_0, and the mean circumference, l, shall be calculated from the equations

$$l_0 = \pi d_\mathrm{i}$$

and

$$l = \pi(d_\mathrm{i} + W_\mathrm{r})$$

where

d_i is the internal diameter;

W_r is the radial width.

9.3 Insertion of test pieces in the fatigue-testing machine

9.3.1 Dumb-bell test pieces

Insert each test piece, in an unstrained state, into the clamps of the test machine. Care shall be taken not to overtighten the clamps, otherwise premature failure might occur at the gripped portion of the test piece. Move the reciprocating part of the machine by hand to the position of maximum extension, and adjust the clamps so that the reference lines on the test pieces are at the required separation. The nominal maximum strain shall not be exceeded during the adjustment. Make a final adjustment 1 min after applying the strain. The measurement shall be made, by Vernier calipers or other means, to an accuracy such that the initial maximum strain is within 2 % (absolute) of the nominal value.

The required separation between the reference lines is given by the formula

$$\left(\frac{e+100}{100}\right) l_0$$

where

e is the required initial maximum strain, expressed as a percentage;

l_0 is the initial unstrained reference length.

For example, for 100 % strain the required distance is twice the initial unstrained reference length.

Move the reciprocating part of the machine to the position of minimum clamp separation and remeasure the reference length. The test piece shall have returned to an unstrained state.

9.3.2 Ring test pieces

Set the machine to the required maximum extension so that a line passing round the periphery of the rollers has the required length to within the accuracy specified for dumb-bells in 9.3.1.

