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Rubber, vulcanized — Determination of tension fatigue

Caoutchouc vulcanisé — Détermination de la fatigue en traction

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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.

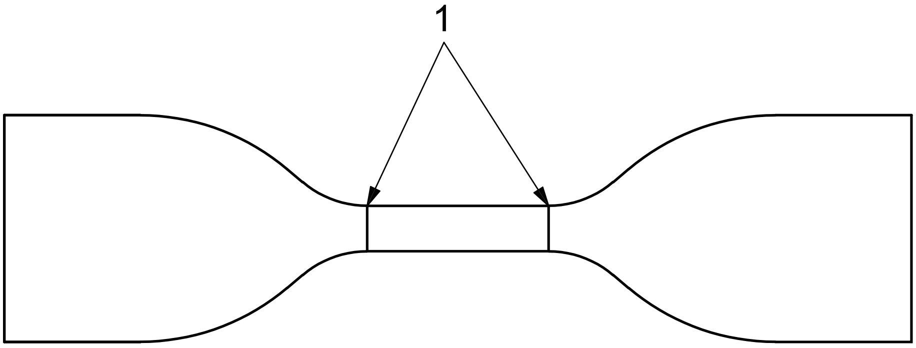
7.1.2 Dumb-bell test piece

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.

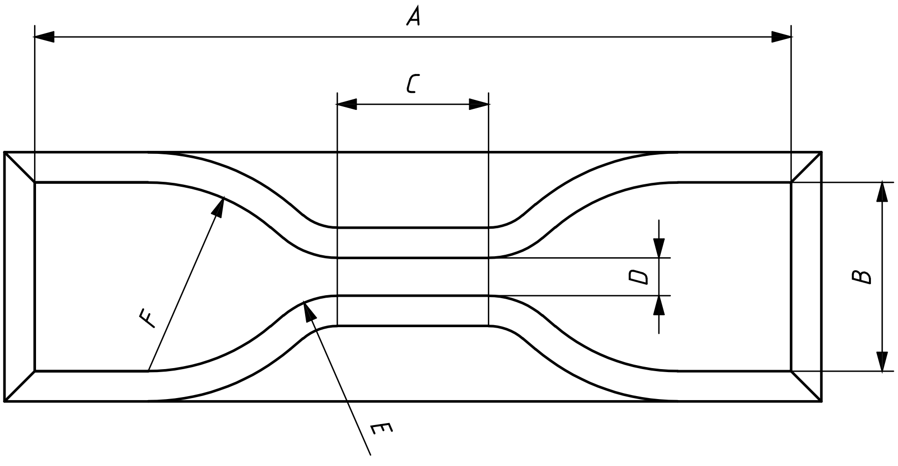
The thickness of dumb-bells shall be (1,5 ± 0,2) mm. In any one dumb-bell, the thickness of the narrow part shall nowhere deviate by more than 2 % from the mean. If results from two sets of dumb-bells are being compared, the mean thicknesses of the sets shall be within 10 % of one another.

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

$$l_0 = \pi d_i$$

and

$$l = \pi(d_i + W_r)$$

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.

Now move the reciprocating part of the machine so that the test piece can be mounted in the unstrained state. The length corresponding to the required maximum strain is given by the formula

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

where l_0 is the initial unstrained internal circumference.

NOTE When the preferred thickness of 1,5 mm is used, the internal diameter of the ring test piece will be very close to that of the cutter. The positions of the rollers of the test machine can thus be calibrated absolutely in this case in terms of strain.

9.4 Determination of fatigue life

When the test pieces have been set up, start the machine and record the number of cycles to break for each test piece.

Alternatively, if a measure of the variability in fatigue life is not required, the test may be terminated before all test pieces have broken, provided sufficient have broken for the calculation of the median fatigue life (see 10.1). It is recommended that, if test pieces remain unbroken after 2×10^6 cycles, the test be terminated unless there is an explicit reason for continuing.

9.5 Measurement of set and maximum strain after cycling

The unstrained length of a test piece increases during a fatigue test because of set. This usually occurs most rapidly at the start of the test and slows down progressively thereafter. If the set is high, the fatigue life can be greatly increased and the results might be misleading. In the case of dumb-bell test pieces, changes can also occur in the maximum extended reference length because of stress softening and set.

Set and the changes in test length shall therefore be determined using a suitable procedure, and the test strains reported in the test report shall be corrected in accordance with 10.3. The set shall not be taken up during the course of the fatigue test.

The method of measurement used shall be reported in the test report. A recommended procedure is as follows:

For each test strain, run two test pieces in the fatigue-testing machine for 1×10^3 cycles and then stop the machine in such a position that one of the test pieces is unstressed. After 1 min, measure the unstrained test length of this test piece. In the case of dumb-bell test pieces, the measurement should be made with the test piece mounted on the machine, which should be moved by hand so that the test piece is just unstressed. In the case of ring test pieces, the test piece should be removed from the machine and measured by means of a cone or other suitable means. Alternatively, the set developed in rings may be measured on the machine by use of automatic force-extension equipment.

Run the machine for another 100 cycles and repeat the procedure just described for the other test piece.

Where necessary, reinsert the test pieces in the machine and repeat the entire procedure after a total of 1×10^4 cycles and after each subsequent decade in the life of the test pieces (i.e. after 1×10^5 cycles, 1×10^6 cycles, etc.).

To measure the change in the maximum extended reference length of dumb-bell test pieces, use the same procedure as used for the determination of set, but with the machine at its maximum separation.

9.6 Measurement of maximum stress and maximum strain energy density

For several purposes, it will be desirable to express fatigue life as a function of the applied maximum stress or the maximum strain energy density (see Annex A). If these parameters are required, it is recommended that stress-strain behaviour be measured both initially and during the course of the fatigue test; like maximum strain, the maximum stress and the maximum strain energy density change during cycling because of the effects of set, stress softening and other factors. If such measurements are made, a separate test piece shall be used for each test material at each test strain.

Automatic force-extension equipment is preferred as this allows changes in maximum force to be followed for both dumb-bell and ring test pieces during the course of the fatigue test, and in the case of ring test pieces it also enables the strain energy density to be obtained at the test frequency.

Alternatively, stress-strain properties may be obtained from a quasi-static force-extension test, either manually or by machine.

The method of measurement shall be reported in the test report. A recommended procedure is as follows:

— Determine the thickness and width of the test piece in accordance with 9.2.

— Use the mean value of each set of measurements in calculating the area of cross-section.

— Extend the test piece to the maximum strain relevant to the particular test, measuring the force-extension behaviour.

Make this measurement either by applying weights to the test piece or by deforming it at a constant rate in a force-measuring machine. If a manual method is used, a regular loading schedule should be adopted, preferably by applying the weights at 1 min intervals and measuring the relevant dimension (the reference length for a dumb-bell test piece or the distance between the rollers for a ring test piece) 30 s after loading. Measure the dimension at suitable increments of strain by Vernier calipers or other means; increments of 10 % to 20 % extension should be suitable for most purposes.

After determining the initial force-extension relationship, insert the test piece in the fatigue-testing machine and cycle at the required test strain to the highest decade of cycling below the median fatigue life (see 10.1) of the test material. (For example, if the test material is found to have a median fatigue life of 6×10^4 cycles, cycle the test piece to 1×10^4 cycles.) Remove the test piece from the machine, remeasure its length, width and thickness after a suitable period of relaxation, and then repeat the force-extension determination.

10 Expression of results

10.1 Calculation of fatigue life

For each test strain, list in ascending order of magnitude the number of cycles to break for each of the fatigued test pieces.

Calculate the median value of the fatigue life and, when appropriate (see Note below), calculate the ratio of the highest to lowest values as a measure of the dispersion. If required, other measures of central tendency and dispersion may be used. These shall be reported in the test report.

Results on any dumb-bell test piece which breaks outside the central narrow section shall be excluded from the calculation of results. However, low results shall not be disregarded unless there is positive, non-statistical evidence that they are unrepresentative (for example, the presence of an abnormally large flaw in the fracture surface clearly attributable to faulty test piece preparation or a foreign body).

For many purposes, it is desirable to plot the results in the form of a graph of fatigue life against maximum strain, stress or strain energy density. It is recommended that a logarithmic scale be used for fatigue life. Strain is generally best plotted on a linear scale. A graph of fatigue life against maximum strain energy density on double-logarithmic scales will often give a linear relationship over a considerable range; a similar graph of fatigue life against maximum stress can also give a linear relationship (although of different slope).

NOTE The inherent variability of fatigue life is large. Both the magnitude of the variability and the nature of the distribution depend on vulcanizate details, particularly the type of rubber that is used. For example, with vulcanizates of natural rubber (NR) or isoprene rubber (IR) the overall variation in life for replicate tests is commonly twofold or less and the distribution often approximates to a normal (Laplace-Gaussian) one; with styrene-butadiene rubber (SBR) or butadiene rubber, on the other hand, the variation can be an order of magnitude or more and the distribution tends to be markedly skew. Because of these differences in behaviour and the complexities they present, particularly in relation to the treatment of blends of different rubbers, it is recommended that simple, generally applicable methods of analysis be used for the statistical treatment of fatigue data.

The median is recommended as a measure of central tendency because it is more representative than the arithmetic mean for rubbers whose lives follow a skewed distribution. Further advantages are that the median is easy to calculate, extreme values are automatically excluded and test time can be saved (at the expense of some loss of precision in estimating dispersion). For rubbers such as NR or IR, six test pieces might give a satisfactory measure of the median, but for SBR and rubbers which behave in a similar way 12 test pieces are likely to be required.

It is important that some measure of dispersion be quoted. A simple measure that has been found useful in fatigue testing is the ratio of highest to lowest lives. In principle, this ratio involves the disadvantage that it does not converge, but for the numbers of test pieces normally used it has been found to correlate closely with the coefficient of variation and is much easier to calculate.

10.2 Calculation of set

The set, expressed as a percentage, developed in test pieces during cycling can be calculated using the formula

$$\left(\frac{l_n - l_0}{l_0}\right)100$$

where, for dumb-bell test pieces:

l_0 is the initial unstrained reference length;

l_n is the unstrained length after the test piece has been fatigued for n cycles;

and, for ring test pieces:

l_0 is the initial unstrained internal circumference;

l_n is the unstrained internal circumference after the test piece has been fatigued for n cycles.

Express the set as the mean value obtained for the two test pieces.

10.3 Calculation of maximum strain

The maximum strain, e_n, expressed as a percentage, corrected for any set that develops during cycling, shall be calculated from the equation

$$e_n = \left(\frac{L_n - l_n}{l_n}\right)100$$

where, for dumb-bell test pieces:

l_n is the unstrained reference length after the test piece has been fatigued for n cycles;

L_n is the distance between the reference lines at maximum clamp separation after the test piece has been fatigued for n cycles (see 9.5);

and, for ring test pieces:

l_n is the unstrained internal circumference after the test piece has been fatigued for n cycles;

L_n is equal to the initial extended length (as this is fixed by the position of the rollers in the test machine, it remains constant during the test).

If required, strain may also be expressed as an extension ratio, λ, which is the ratio of the extended reference length or internal circumference to the unstrained reference length or internal circumference. Thus a strain of 75 % elongation is equivalent to a λ of 1,75.

10.4 Calculation of maximum stress

Calculate the maximum stress, in megapascals, by dividing the maximum force, in newtons, by the unstressed cross-sectional area, in square millimetres (for dumb-bell test pieces) or by twice the unstressed cross-sectional area, in square millimetres (for ring test pieces).

For the calculation of maximum stress after a period of cycling, the unstressed cross-sectional area and the relevant maximum strain shall be determined from the dimensions of the cycled test piece. If the change in the unstressed cross-sectional area is small after the period of cycling, the dimensions before cycling may be used instead.

10.5 Calculation of strain energy density

If the quasi-static force-extension method described in 9.6 has been used, plot curves of stress against strain using the values obtained before and after the period of cycling. From each of these curves, calculate, by means of numerical integration, the strain energy density as a function of the maximum strain. The strain energy density or strain energy per unit volume is equal to the area under the stress-strain curve. It shall be expressed in joules per cubic metre.

If the force-extension behaviour is measured automatically for ring test pieces, calculate the maximum strain energy density at the test frequency by dividing the area under the extension part of the force-extension curve by the test piece volume. The test piece volume shall be taken as the mean circumference, l (see 9.2.2), multiplied by the cross-sectional area.

11 Test report

The test report shall contain the following information:

a) sample details:

   1) a full description of the sample and its origin,

   2) the method of preparation of test piece from the sample, for example whether moulded or cut or taken from finished products;

b) a full reference to the test method used, i.e. the number of this International Standard;

c) test details:

   1) the type of test piece used, i.e. dumb-bell (type 1, type 1A or type 2) or ring,

   2) the frequency of cycling,

   3) the temperature of test,

   4) the type of atmosphere used (if different from that specified in 8.4),

   5) the method used to calculate the set developed during cycling,

   6) the method used to calculate the maximum stress and the strain energy density when these are required,

   7) the number of test pieces used,

   8) details of any procedures not specified in this International Standard;

d) test results:

   1) for each test strain, the individual fatigue lives, the median fatigue life and, if measured, the ratio of the highest to lowest fatigue life,

   2) the initial test strain or test strains,

3) for each test strain, the mean value of the set developed and the number of cycles after which the set was measured,

4) the test strains corrected for set, and the number of cycles after which they were measured,

5) if required, the maximum stress and the strain energy density in the test pieces, and the number of cycles after which they were measured,

6) if required, graphs of median fatigue life against maximum strain, stress or strain energy density determined before and/or after periods of cycling;

e) the date of the test.

Annex A
(informative)

Explanatory notes

A.1 Introduction

The basic process involved in tension fatigue is crack growth initiated by flaws that create stress concentrations in the rubber. Fracture mechanics studies have shown that the crack growth behaviour of a vulcanizate is independent of test piece shape and type of deformation if it is related to the elastic energy available for the propagation of a crack[1][2]. This relation is thus a fundamental property of a vulcanizate — its crack growth characteristics — which can, in principle, also be used to relate laboratory tests to service performance. The crack growth characteristics can be determined by laboratory measurements on certain test pieces of simple shape, but such measurements present various difficulties from a routine test viewpoint; the De Mattia method (see ISO 132) is not suitable for determining the crack growth characteristics.

Tension fatigue tests, although less fundamental, offer a straightforward alternative to basic crack growth measurements. Fatigue results can, if desired, be used to calculate the crack growth characteristics provided the initial flaw size is known.

A.2 Factors affecting fatigue life

Fatigue is influenced by many variables, which can be grouped into three classes. First, those representative of the material: the type of rubber, the type and degree of crosslinking, the presence of additives such as fillers and protective agents (which govern the basic crack growth characteristics) and the size of the flaws that are present initially. Second, mechanical aspects: the shape and size of the article, the nature and magnitude of the deformations and the frequency and form of cycling. Third, environmental factors: temperature, humidity and the presence of potentially hostile agents such as atmospheric oxygen and ozone.

Primary causes of crack growth and fatigue in natural environments are the mechanical stresses, oxygen and ozone. The relative effects of these are illustrated in Figure A.1, which shows results for an unfilled natural-rubber vulcanizate containing 1 pphm of antioxidant in various atmospheres. The results *in vacuo* represent purely mechanical failure; it can be seen that the life becomes very long (it is believed to become virtually infinite) as the maximum strain of the fatigue cycle is reduced towards 100 %. The latter strain thus constitutes a mechanical-fatigue limit, below which no failure occurs in the absence of chemical attack. A similar fatigue limit is observed for other rubbers, although its magnitude can vary.

The results in the laboratory atmosphere illustrate the influence of oxygen, which is to reduce the fatigue limit — to about 75 % in the present case — and to reduce the life at higher strains. These reductions are greater if the rubber contains no antioxidant (although the life *in vacuo* is unaffected); thus the antioxidant is partially, but not wholly, successful in mitigating the effects of oxygen. Failure at strains below the reduced fatigue limit is initiated by attack by the small quantity of ozone present in the laboratory atmosphere.

The ozone chamber results show that the ozone concentration is important below the fatigue limit but has little effect at higher strains. All the results illustrate the strong dependence of the fatigue life on the maximum strain.

A.3 Choice of test piece

Essentially the same results should normally be obtained with either dumb-bell or ring test pieces, provided they are cut properly; however, if the intrinsic flaw size of a particular vulcanizate is sensitive to a test piece size within the range involved, fatigue lives for dumb-bells can be consistently higher than for rings (owing to the smaller volume of rubber in the test section of the former test pieces). Use of dumb-bells is essential for studying directional effects. Rings are very easy to set up, however, and the machine setting for a given initial strain cycle can be calculated independently of the rubber. In addition, stresses are essentially uniform around the circumference of a ring test piece (and with the dimensions specified vary only slightly across the width) so that stress-strain behaviour and set can be monitored automatically, enabling changes in these properties to be followed during the test.

A.4 Interpretation of fatigue results

If different elastomers are being compared, their ranking order could well vary depending on the maximum strain employed; it is therefore important to carry out measurements at various strains and to try to identify the conditions relevant for a particular application. Where only minor changes in formulation are made, such as in evaluating different protective systems, fatigue measurements at a single strain might be adequate provided the stress-strain behaviour does not vary significantly.

For articles that have long lives under repeated deformations of fairly constant amplitude through zero strain, tests in the vicinity of the fatigue limit are likely to be appropriate. The life at the fatigue limit is generally of the order of 10^5 cycles to 10^6 cycles and increases rapidly with decreasing strain; thus the strain corresponding to the latter life often gives a reasonable estimate of the fatigue limit. For articles that are shorter-lived, or are subject to occasional large deformations superimposed on many smaller ones, severities well above the fatigue limit could be appropriate. In some instances, both aspects of behaviour might be relevant. For tyre treads, for example, groove cracking is affected by properties in the fatigue limit region[3], while rib-tearing probably depends on high-severity crack growth and tear resistance.

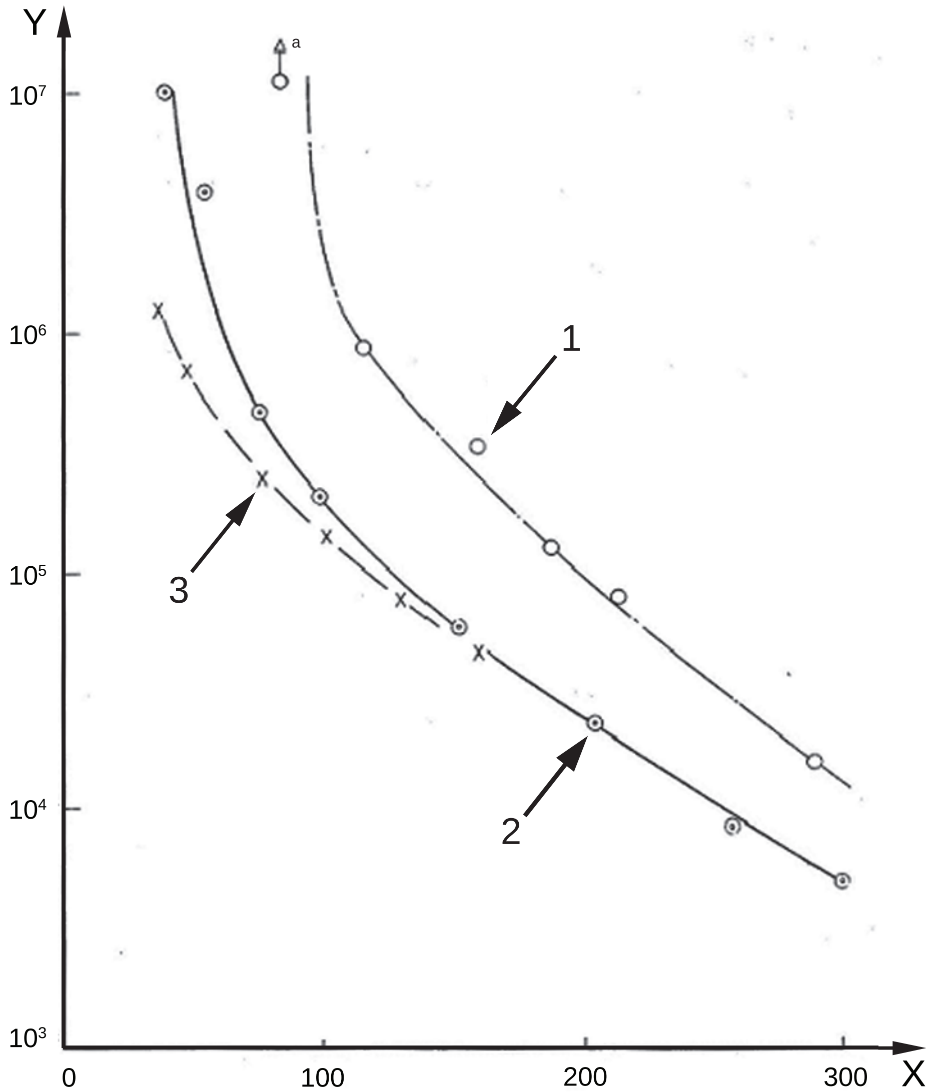
The basis on which results should be compared — constant strain, constant stress or constant strain energy density — depends on the conditions applying in service. If the latter are difficult to assess, it might be best to compare results by more than one method.

Strain energy density is the elastic energy stored per unit volume in the bulk of the deformed rubber and, together with flaw or crack size, determines the magnitude of the fundamental tearing energy at the tip of the crack. Thus, comparison at constant strain energy density eliminates any effects due to differences in modulus or stress-strain response.

Use of appropriate atmospheres and temperatures is also recommended, since the response of different materials to the environment varies — for example, the temperature dependence of fatigue life is generally much stronger for non-crystallizing than for crystallizing elastomers. Results for materials that show considerable stress softening or set, or highly viscous behaviour, should be interpreted with caution; this applies particularly at high temperature, since changes in stress-strain behaviour and set can be very marked, even for materials that are well behaved at room temperature.

It is important that the variability of results be assessed, not only as an indication of possible variations in service performance but also as a check on faulty test piece preparation or test procedure. The dispersion varies for different materials, but each has its own characteristic behaviour. For samples of six to 12 replicate tests, the ratio of highest to lowest lives does not normally exceed about 2 for vulcanizates of NR or IR, or about 10 for vulcanizates of SBR or BR. Variability well in excess of that expected is a good indication that something has gone wrong. In particular, anisotropy or built-in stresses in the rubber can cause very marked variations in crack growth and fatigue behaviour — much more so than in other properties — and can considerably confuse interpretation of the results.

For example, for an anisotropic sheet which showed a variation in Young's modulus of about 20 % in different directions, the variation in crack growth resistance was found to be more than a thousandfold.



Key

X maximum strain (%)

Y fatigue life (No. of cycles)

1 *in vacuo*

2 laboratory atmosphere (ozone concentration about 0,2 pphm)

3 ozone chamber (ozone concentration 8 pphm)

[a] Test piece removed unbroken.

Figure A.1 — Effects of maximum strain and atmosphere on tension fatigue life (natural-rubber vulcanizate)

Annex B
(normative)

Calibration schedule

B.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded in any calibration report or certificate. It shall be reported whether calibration is carried out in the “as-received” condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to charge, then the need for periodic checks shall be written into the detailed calibration procedures.

B.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this International Standard. However, the frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table B.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that specified in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code-letter. The code-letters used in the calibration schedule are:

C requirement to be confirmed, but no measurement;

N initial verification only;

S standard interval as given in ISO 18899;

U in use.

In addition to the items listed in Table B.1, use of the following is implied, all of which need calibrating in accordance with ISO 18899:

— a timer;

— a thermometer for monitoring the conditioning and test temperatures;

— instruments for determining the dimensions of the test pieces.

Table B.1 — Calibration frequency schedule

Parameter	Requirement(s)	Subclause in ISO 18899:2004	Verification frequency guide	Notes
Fatigue-testing machine	Frequency of reciprocating motion 1 Hz to 5 Hz	23.3	S	
	Stroke adjustable	C	N	
	Motor does not generate ozone	C	N	
Grips	For dumb-bell test pieces: no slippage	C	U	
	For ring test pieces: rollers as specified in 5.1	C	N	
Dies and cutters	In accordance with ISO 23529	C	N	
	Dimensions of dies for dumb-bells as specified in Table 1	15.2, 15.3, 15.9	S	
Marker for dumb-bells	Width at edges 0,05 mm to 0,10 mm	15.2	S	
	Edges bevelled at ≤ 15°	15.9	S	
Marking substance	As specified in 5.4	C	U	
Dial gauge	In accordance with ISO 23529	15.1, 16.6	S	
Vernier calipers or travelling microscope	Accurate to ±0,2 mm	15.1	S	See also ISO 23529
Graduated cone	Accurate to ±0,2 mm	15.1	S	See also ISO 23529

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[9] ISO 4666-4, *Rubber, vulcanized — Determination of temperature rise and resistance to fatigue in flexometer testing — Part 4: Constant-stress flexometer*

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