INTERNATIONAL STANDARD

ISO 132

Third edition
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Rubber, vulcanized or thermoplastic — Determination of flex cracking and crack growth (De Mattia)

Caoutchouc vulcanisé ou thermoplastique — Détermination de la résistance au développement d'une craquelure (De Mattia)

Reference number
ISO 132:1999(E)

Content

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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.

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.

ISO 132 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

This third edition cancels and replaces the second edition (ISO 132:1983) and ISO 133:1983, of which it constitutes a technical revision.

Introduction

Repeated bending or flexing of a rubber causes cracks to develop in that part of the surface where tension stress is set up during flexing or, if this part of the surface contains a crack, causes this crack to extend in a direction perpendicular to the stress. Certain soft vulcanizates, for instance those prepared from styrene-butadiene rubber, show marked resistance to crack initiation, but it is possible for these vulcanizates to have a low resistance to growth (propagation) of cracks. It is important, therefore, to measure both the resistance to crack initiation by flexing and the resistance to crack propagation.

The method is suitable for rubbers that have reasonably stable stress-strain properties, at least after a period of cycling, and do not show undue stress softening or set, or highly viscous behaviour. The results obtained for some thermoplastic rubbers should be treated with caution if the elongation at yield is below, or close to, the maximum strain imposed during the test.

Rubber, vulcanized or thermoplastic — Determination of flex cracking and crack growth (De Mattia)

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

1 Scope

This International Standard specifies a method of test intended for use in comparing the resistance of rubbers to the formation and growth of cracks, when subjected to repeated flexing on the De Mattia type machine. For determination of crack growth, an artificial cut is made in the test piece to initiate cut growth.

2 Normative references

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

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

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

ISO 4661-1:1993, *Rubber, vulcanized or thermoplastic — Preparation of samples and test pieces — Part 1: Physical tests.*

3 Apparatus

3.1 De Mattia type machine.

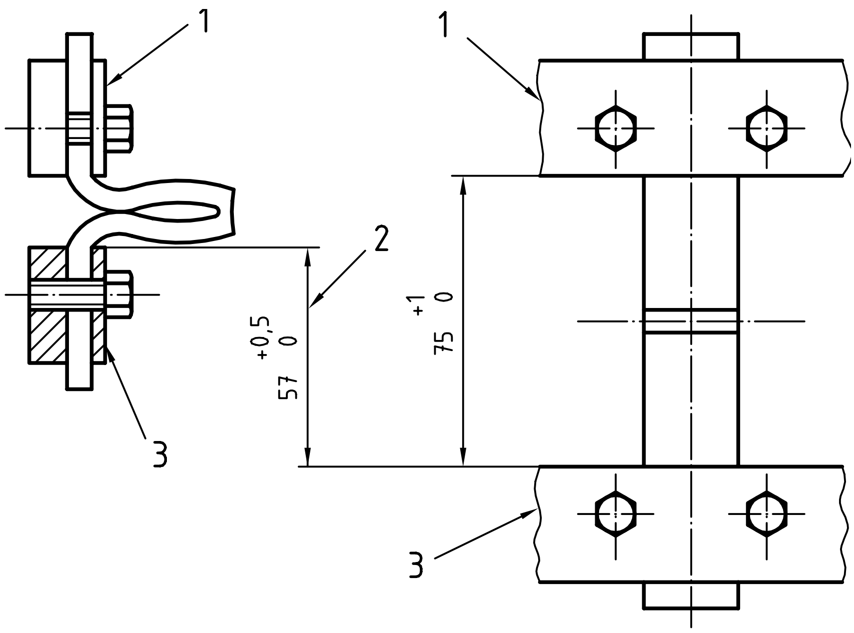
The essential features of the De Mattia type machine are as follows:

Stationary parts, provided with grips for holding one end of each of the test pieces in a fixed position, and similar but reciprocating parts for holding the other end of each of the test pieces. The travel is ($57^{+0,5}_{0}$) mm and is such that the maximum distance between each set of opposing grips is (75^{+1}_{0}) mm (see figure 1).

The reciprocating parts are so arranged that their motion is straight and in the direction of, and in the same plane as, the common centreline of each opposing pair of grips. The planes of the gripping surfaces of each opposing pair of grips remain parallel throughout the motion.

The eccentric which actuates the reciprocating parts is driven by a constant-speed motor to give 5,00 Hz ± 0,17 Hz, with sufficient power to flex at least six, and preferably twelve, test pieces at one test. The grips hold the test pieces firmly, without undue compression, and enable individual adjustment to be made to the test pieces to ensure accurate insertion.

Dimensions in millimetres



Key

1 Upper grip

2 Travel

3 Lower grip

Figure 1 — De Mattia type machine

It is useful to arrange the test pieces in two equal groups, so that one group is being flexed while the other group is being straightened, thus reducing the vibration in the machine.

For testing at elevated or subnormal temperatures, the machine may be enclosed in a chamber with temperature control near the centre of the test piece to ± 2 °C, if necessary, by using an air circulator.

3.2 Piercing tool and suitable jig, for piercing the test pieces (see 4.2).

4 Test pieces

4.1 Shape, dimensions and preparation

Each test piece shall be a strip with a moulded groove, as shown in figure 2. The strips may be moulded individually in a multiple-cavity mould or may be cut from a wide slab having a moulded groove.

The groove in the test piece shall have a smooth surface and be free from irregularities from which cracks may start prematurely. The groove shall be moulded into the test piece or slab by a half-round ridge in the centre of the cavity. The half-round ridge shall have a radius of 2,38 mm ± 0,03 mm. The moulded groove shall be perpendicular to the direction of calendering.

Results may be compared only between test pieces having thicknesses, measured close to the groove, which are within the tolerances, because the results of the test are dependent upon the thickness of the test piece.

If finished products are to be tested, test pieces without a groove can be used. They shall be prepared in accordance with ISO 4661. Cracks shall not be assessed on surfaces that have been cut or buffed. The use of test pieces cut and/or buffed from finished products shall be stated in the test report.

Dimensions in millimetres

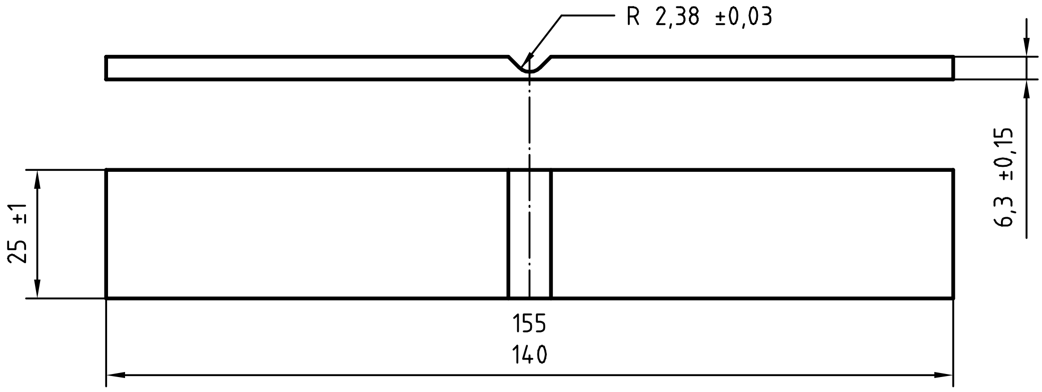


Figure 2 — Test piece

4.2 Preparation of test pieces for cut growth measurement

Each test piece shall be prepared by piercing the bottom of the groove at a point equidistant from the sides, using a suitable jig. The piercing tool shall conform to the dimensions given in figure 3. The piercing tool shall be maintained perpendicular to both the transverse and longitudinal axes, and the cut accomplished by a single insertion and withdrawal of the tool. The cut shall be parallel to the longitudinal axis of the groove. Lubrication with water containing a suitable wetting agent may be used.

A suitable jig shall be provided to hold the cutting tool; the exact details are not specified but the principles of operation shall be as follows:

The test piece shall be held flat in a solid support. The cutting tool shall be normal to the support and placed centrally with respect to the groove in the test piece, with the cutting edge of the piercing tool parallel to the axis of the groove. Means shall be provided for passing the piercing tool through the entire thickness of the rubber, and the support shall have a hole of a size just sufficient to permit the piercing tool to project through the base of the test piece to not less than 2,5 mm and not more than 3 mm.

4.3 Time interval between vulcanization and testing

For all test purposes, the minimum time between vulcanization and testing shall be 16 h in accordance with ISO 471.

For non-product tests, the maximum time between vulcanization and testing shall be 4 weeks and, for evaluations intended to be comparable, the tests shall, as far as possible, be carried out after the same time interval.

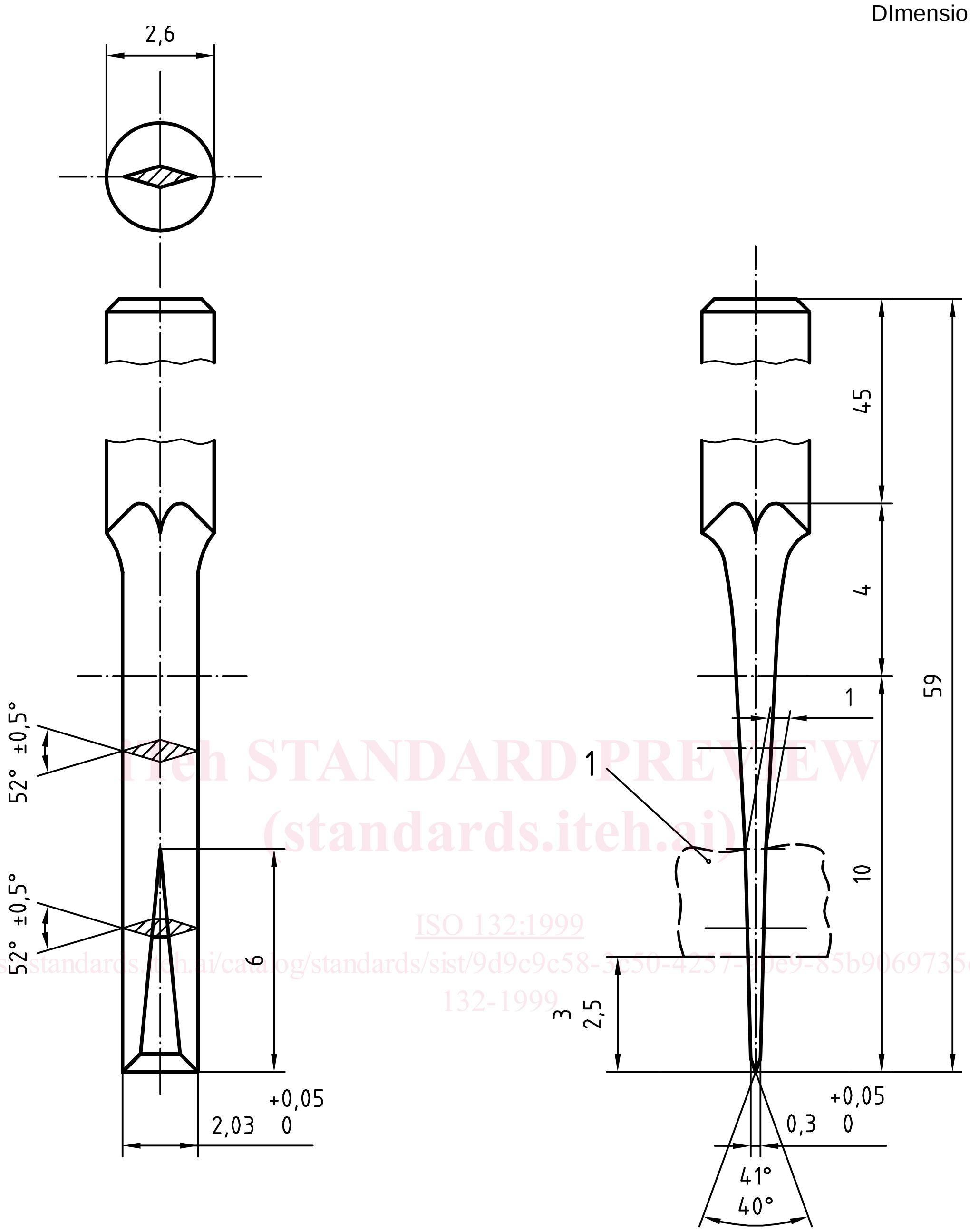
As far as possible, samples and test pieces shall be kept away from exposure to light.

4.4 Conditioning

For tests under standard laboratory conditions (see clause 5), individually moulded test pieces shall be conditioned under the test conditions during a period of time in accordance with ISO 471 immediately before testing.

Slab samples shall be similarly conditioned before the test pieces are cut. These test pieces may be either tested immediately or kept at the test temperature until tested.

Dimensions in millimetres



Key

1 Test piece

Figure 3 — Piercing tool

For tests at other temperatures (see clause 5), after the conditioning period specified above the test pieces shall be brought to the test temperature by keeping in a chamber at this temperature for 3 h, then tested immediately (see ISO 3383).

The same test temperature shall be used throughout any test or series of tests intended to be comparable.

4.5 Number of test pieces

At least three, and preferably six, test pieces from each rubber compound shall be tested.

If comparison is required between different compounds, ensure that test pieces of each compound are mounted at the same time on the same test machine.

5 Test conditions

5.1 Temperature

Tests are normally performed at a standard laboratory temperature as defined in ISO 471, although elevated or subnormal temperatures may often be used to advantage. In the latter case, the test temperature shall be selected from ISO 471.

5.2 Humidity

With compounds where the results are known to be sensitive to humidity, the test shall be carried out under standard laboratory conditions (temperature and humidity) as defined in ISO 471.

NOTE An influence of humidity has been observed with fluororubbers, polyurethane rubbers and other rubbers containing hydrophilic fillers.

6 Procedure

6.1 General

The presence of significant amounts of ozone in the laboratory atmosphere affects the results. Periodic checks are advised to ensure that the ambient partial pressure of ozone is preferably less than 1 mPa.

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

6.2 Determination of flex cracking

Separate the pairs of grips to their maximum extent, and insert the test pieces so that they are flat and not under tension, with the groove in each test piece midway between the two grips in which that test piece is held, and on the outside of the angle made by the test piece when it is bent.

Ensure that the test pieces are positioned at exactly 90° to the grips.

Start the machine and continue the test with frequent inspections until the first small sign of cracking is detected on each test piece. Record the number of flexing cycles at this point, restart the machine and stop it after intervals of, for instance, 1 h, 2 h, 4 h, 8 h, 24 h, 48 h, 72 h and 96 h. As an alternative, stop it after intervals in which the number of flexing cycles is increased in geometric progression, a suitable ratio being 1,5 on each occasion. Carry out each inspection of the test pieces with the test pieces fixed in place but the grips separated to a distance of 65 mm.

Cracks occurring at the edge of the test piece shall be ignored.

Grade the severity of cracking comparison with a standard scale, as specified in 7.1. Do not run the test pieces to complete rupture, but to a given grade of cracking.

6.3 Determination of crack growth

Measure the initial length L of the cut, preferably using a low-power magnifying glass.

Insert the test pieces as described in 6.2, first paragraph.

Start the machine and stop it at frequent intervals to measure the length of the cut, for example after 1, 3 and 5 kilocycles and at such further or intermediate intervals as appear necessary. At each inspection, separate the grips to a distance of 65 mm and measure the length of the cut, preferably using a low-power magnifying glass.

Do not run the test pieces to complete rupture, but to a specified crack growth in accordance with 7.2.

7 Expression of results

7.1 Determination of flex cracking

The comparison includes an assessment of the length, depth and number of cracks.

Cracking shall be graded in accordance with the following scale:

Grade 1

The cracks at this stage look like pin pricks to the naked eye. Grade as 1 if the “pin pricks” are 10 or less in number.

Grade 2

Assess as grade 2 if either of the following applies:

a) the "pin pricks" exceed 10 in number;

b) the number of cracks is less than 10, but one or more cracks have developed beyond the "pin prick" stage, i.e. have perceptible length without much depth and their length is not more than 0,5 mm.

Grade 3

One or more of the "pin pricks" have become obvious cracks, i.e. have appreciable length and little depth and their length is greater than 0,5 mm but not greater than 1 mm.

Grade 4

The length of the largest crack is greater than 1 mm but not greater than 1,5 mm.

Grade 5

The length of the largest crack is greater than 1,5 mm but not greater than 3 mm.

Grade 6

The length of the largest crack is greater than 3 mm.

NOTE No distinction is made between cracks that have grown in isolation and those that have grown by coalescence.

Calculate the median number of kilocycles to reach each grade of cracking. Plot the grades from 1 to 6 against the median number of corresponding kilocycles of flexing on linear graph paper and draw a smooth curve through the points. Using graphical interpolation, deduce the number of kilocycles required for each grade of cracking.

The number of kilocycles required to reach grade 3 is the mean flex cracking resistance.

Instead of graphical interpolation, a computer programme can be used for the calculation.

7.2 Determination of crack growth

Plot the crack length versus the number of flexing cycles for each test piece. Draw a smooth curve through the points and read off

a) the number of kilocycles required for the cut to extend from L mm to (L + 2) mm;

b) the number of kilocycles required for the cut to extend from (L + 2) mm to (L + 6) mm;

c) if desired, the number of kilocycles required for the cut to extend from (L + 6) mm to (L + 10) mm.

For each of these cut extensions, calculate the median number of kilocycles.