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Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression at ambient and at elevated temperatures

Caoutchouc vulcanisé ou thermoplastique — Détermination de la relaxation de contrainte en compression à température ambiante et aux températures élevées

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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.

International Standard ISO 3384 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Sub-Committee SC 2, *Physical and degradation tests*.

This third edition cancels and replaces the second edition (ISO 3384:1986), of which it constitutes a technical revision.

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Introduction

When a constant strain is applied to rubber, the force necessary to maintain that strain is not constant but decreases with time; this behaviour is called "stress relaxation". Conversely, when rubber is subjected to a constant stress, an increase in the deformation takes place with time; this behaviour is called "creep".

The processes responsible for stress relaxation may be physical or chemical in nature, and under all normal conditions both types of process will occur simultaneously. However, at normal or low temperatures and/or short times, stress relaxation is dominated by physical processes whilst at high temperatures and/or long times chemical processes are dominant. Hence it is neither safe to extrapolate time/stress relaxation curves in order to predict stress relaxation after periods considerably longer than those covered by the test, nor to use tests at higher temperatures as accelerated tests to give information on stress relaxation at lower temperatures.

In addition to the need to specify the temperatures and time intervals in a stress relaxation test, it is also necessary to specify the initial stress and the previous mechanical history of the test piece since these may also influence the measured stress relaxation, particularly in rubbers containing fillers.

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Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression at ambient and at elevated temperatures

1 Scope

This International Standard specifies three methods for determining the decrease in counterforce exerted by a test piece of vulcanized or thermoplastic rubber which has been compressed to a constant deformation and maintained thus at a predetermined test temperature.

Two forms of test piece are permitted: cylindrical test pieces and rings. Different shapes and sizes of test piece give different results, and comparison of results should be limited to test pieces of similar size and shape.

The use of ring test pieces is particularly suitable for the determination of stress relaxation in liquid environments.

Testing at temperatures below standard temperature is not specified.

The methods have been used for low-temperature testing, but their reliability under these conditions is not proven.

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 37:1977, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 468:1982, *Surface roughness — Parameters, their values and general rules for specifying requirements.*

ISO 471:1983, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1817:1985, *Rubber, vulcanized — Determination of the effect of liquids.*

ISO 1826:1981, *Rubber, vulcanized — Time-interval between vulcanization and testing — Specification.*

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

ISO 3601-1:1988, *Fluid systems — Sealing devices — O-rings — Part 1: Inside diameters, cross-sections, tolerances and size identification code.*

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

ISO 4661-1:1986, *Rubber, vulcanized — Preparation of samples and test pieces — Part 1: Physical tests.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 compression stress relaxation: The reduction in compressive force, expressed as a percentage of the initial force, which occurs with time after the application of a constant compressive strain.

4 Principle

A test piece of vulcanized or thermoplastic rubber is compressed to a constant deformation and maintained at a pre-determined test temperature. The decrease in counterforce that is exerted is then measured.

In method A, the compression is applied and all counterforce measurements are made at the test temperature.

In method B, the compression is applied and all counterforce measurements are made at standard temperature. The test pieces are stored at the test temperature.

In method C, the compression is applied at standard temperature and all counterforce measurements are made at the test temperature.

NOTE 1 The three methods A, B and C of carrying out the measurement do not give the same values of stress relaxation, and comparison of values obtained from the three methods should be avoided. The method selected for use depends on the purpose of the test. Thus, for fundamental studies and in applications where sealing at elevated temperatures is a problem, method A or C may be preferred, and in applications where temperature cycling from normal to an elevated temperature is a problem, method B may be preferred.

5 Apparatus

5.1 Compression device, consisting of two parallel, flat, highly polished plates made from chromium-plated or stainless steel or other corrosion-resistant material, between the faces of which the test pieces are compressed.

Flatness, surface roughness, parallelism and rigidity of the plates are all important.

When the apparatus is disassembled, the compression plates shall be flat to within 0,01 mm. The finish of the surface shall not be worse than 0,4 µm Ra (see ISO 468). When the apparatus is assembled without a specimen test piece, the gap between the plates shall not vary by more than ± 0,01 mm.

When the test assembly is subjected to the test load with a test piece between the plates, neither compression plate shall bend by more than 0,01 mm.

The plates shall be of sufficient size to ensure that the whole of the compressed test piece is within the area of the plates.

For ring test pieces, the plates shall have holes of at least 2 mm diameter drilled through their centre portions to allow equalization of pressure and circulation of fluid inside the ring-shaped test piece.

It shall be possible to connect the compression device to suitable equipment for compressing the test piece to the specified compression at the specified speed and for measuring the counterforce exerted by the compressed test piece with an accuracy of 1 % of the measured value.

The device shall be capable of setting and maintaining the compression during the whole duration of the test and it shall be possible to keep it in an oven at the specified test temperature. Care shall be taken to ensure that there is no loss of heat from the test piece, for example by conduction through metal parts which are connected with the outside of the oven.

5.2 Counterforce-measuring device, capable of measuring compression forces in the desired range with an accuracy of 1 % of the measured value. The device may be one that monitors the test piece during the whole duration of test, in which case a continuous measurement of the change of counterforce with time is possible. The stiffness of the counterforce measuring device shall be sufficient for the change in compression of the test piece due to the relaxation of the load to be less than 0,02 mm.

Alternatively, a compression testing machine may be used to measure the counterforce at prescribed time intervals. In this case, the force necessary to cause a slight increase in the compression of the test piece is measured. This additional compression shall be as small as possible and in no case greater than a force of 1 N for balance-type machines, or greater than 0,05 mm for stress/strain-type machines, applied in either case without overshoot.

5.3 Test chamber, complying with the requirements of ISO 3383.

For tests in air, a well designed, uniformly heated air oven shall be used, provided with adequate temperature control to maintain the specified air temperatures within the tolerances specified in 8.2. Satisfactory circulation of the air shall be secured by means of a fan.

For tests in liquids, the compression device shall be totally immersed in the liquid in a bath, or a closed vessel for volatile and toxic fluids, such that free circulation of the liquid can take place through the holes in the compression plates. The liquid shall be maintained at the specified temperature by proper control of a heater and circulation of the liquid in the bath or, alternatively, by placing the liquid bath and compression device within an air oven as specified above.

5.4 Temperature-measuring equipment, with a sensing element, for example a thermocouple. The thermo-measuring element shall be mounted so that it is located no more than 2 mm from a surface of the test piece.

6 Test piece

6.1 Type and preparation of test piece

6.1.1 General

Test pieces may be prepared either by moulding or in accordance with ISO 4661-1, by cutting from moulded sheets or products.

6.1.2 Cylindrical test pieces

The test piece shall be a cylindrical disc of diameter 13 mm ± 0,5 mm and thickness 6,3 mm ± 0,3 mm or of diameter 29,0 mm ± 0,5 mm and thickness 12,5 mm ± 0,5 mm. The smaller test piece is preferred.

6.1.3 Ring test pieces

The preferred test piece is a ring of square cross-section, cut from a flat sheet of the test material by means of rotary cutters. For a suitable machine for the preparation of small ring test pieces, see the annex of ISO 37:1977.

The dimensions of test pieces shall be:

- thickness: 2,0 mm ± 0,2 mm
- inner diameter: 15,0 mm ± 0,2 mm
- radial width: 2,0 mm ± 0,2 mm

The sheets may be prepared by moulding or from finished articles by cutting and buffing.

Alternatively, an O-ring, size code B0140G as specified in ISO 3601-1 (diameter of the cross-section 2,65 mm and internal diameter 14,0 mm), may be used as the standard test piece.

O-rings of other dimensions, together with seals or gaskets of other configurations, may be used as non-standard test pieces where appropriate.

Most test machines have jigs in which the test piece is compressed by screwing a compression plate down to stops. This gives a fixed, strained thickness. Test pieces within the tolerances given above will not necessarily have the required compression strain when tested in such jigs. It is important that a compression strain within the limits given in 8.3.4, 8.4.3 or 8.5.3 is achieved by careful matching of jig and test piece.

NOTE 2 The results obtained from the different types of test piece cannot be compared.

6.2 Measurement of dimensions of test pieces

The dimensions of the test pieces shall be measured as specified in ISO 4648.

6.3 Number of test pieces

At least two tests using separate test pieces shall be carried out.

6.4 Time interval between vulcanization and testing

The interval between vulcanization and testing shall be in accordance with ISO 1826.

6.5 Conditioning of test pieces

6.5.1 For many materials, and particularly for compounds containing substantial proportions of filler, reproducibility of results may be improved by mechanically conditioning the test piece followed by thermal conditioning. This shall be carried out as specified in 6.5.2. Alternatively, thermal conditioning only shall be carried out as specified in 6.5.3.

6.5.2 Mechanical conditioning should only be applied when it is relevant to the final application. When used, it shall be applied at one of the standard temperatures specified in ISO 471, as follows:

Compress the test piece to the same strain that will be used during the rest of the test and then immediately return it to zero deformation; repeat this procedure to give a total of five cycles of deformation and immediate return.

NOTE 3 Thermoplastic elastomers may contain moulding stresses, and thermal conditioning to relieve them will improve reproducibility of results. For some thermoplastic elastomers, a conditioning of 3 h at 70 °C has been found suitable.

Follow any mechanical conditioning by thermal conditioning for a period of not less than 16 h and not more than 48 h at standard temperature immediately before testing.

6.5.3 Thermal conditioning of test pieces which have not been subjected to mechanical conditioning shall consist of maintaining the test pieces at one of the standard temperatures for a period of 3 h immediately before testing.

7 Duration, temperature and test liquid

7.1 Duration of test

The preferred duration of test is ($168\ ^{0}_{-2}$) h.

If intermediate times are used, 3 h $^{0}_{-10}$ min, 6 h $^{0}_{-20}$ min, (24 $^{0}_{-0,5}$) h, and (72 $^{0}_{-1}$) h are preferred. The test period begins after the initial compression. If longer testing times are needed, a logarithmic time-scale shall be used.

In method B, when compression is carried out at standard temperature, each time the test piece is conditioned for measurement at that temperature, a conditioning period of 2 h (not included in the time of test) shall be allowed.

7.2 Temperature of exposure

The temperature of exposure shall be chosen from one of the following list of preferred temperatures (see ISO 471):

23 °C ± 2 °C
27 °C ± 2 °C
40 °C ± 1 °C
55 °C ± 1 °C
70 °C ± 1 °C
85 °C ± 1 °C
100 °C ± 1 °C
125 °C ± 2 °C
150 °C ± 2 °C
175 °C ± 2 °C
200 °C ± 2 °C
225 °C ± 2 °C
250 °C ± 3 °C

If there is no technical reason for choosing a particular temperature, 100 °C shall be used.

Temperatures of exposure which cause rapid degradation or evaporation of the test liquid shall be avoided.

7.3 Immersion liquids

The test liquid shall be chosen according to the particular application, but should preferably be one of those listed in ISO 1817.

8 Procedure

8.1 Preparation

Carefully clean the operating surfaces of the compression device. When a lubricant is applied, it shall consist of a thin coating of a lubricant having substantially no action on the rubber. For most purposes, a silicone or fluorosilicone fluid is suitable.

8.2 Thickness measurement

8.2.1 Cylindrical test pieces

Measure the thickness of each test piece at the central portion with an accuracy of 0,01 mm after conditioning at the chosen standard temperature as specified in ISO 4648. The thickness shall be determined in accordance with ISO 4648:1991, method A.

8.2.2 Ring test pieces

Measure the axial thickness of each test piece with an accuracy of 0,01 mm at four points approximately 90° apart around the ring, at the chosen standard temperature, as specified in ISO 4648. The average of the measurements shall be used for calculation of the necessary compression. Individual measurements on a single test piece shall not differ by more than 0,05 mm; if they do discard the test piece.

8.3 Method A

8.3.1 Bring the compression device and the test environment to the test temperature.

8.3.2 When testing in a liquid environment, the test piece and the operating surfaces of the compression device shall be gently lubricated with the test liquid. When testing in a gaseous medium, a thin coating of a lubricant having substantially no action on the rubber shall be applied. A silicone or fluorosilicone fluid, having a kinematic viscosity of about 0,01 m^2/s, and molybdenum disulfide, have been found to be suitable lubricants.

8.3.3 Immediately after lubrication, condition the test piece at the test temperature in accordance with ISO 3383. Conditioning for 30 min is recommended. For temperatures upwards of 150 °C, longer times are necessary in accordance with ISO 3383.

8.3.4 Compress the preheated test piece by (25 ± 2) % in the compression device (5.1) at the test temperature; use a compression of (15 ± 2) % or lower, in steps of 5 %, if a compression of 25 % cannot be obtained. Compress the test piece in a time between 30 s and 2 min. When reached, the final compression shall be fixed and maintained during the entire test period (apart from the further small compression which may be used for measurement of counterforce as mentioned in 5.2).

8.3.5 Within 5 min of completing the compression, immerse the part of the compression device containing the test piece in the test environment at the test temperature.

8.3.6 Measure the counterforce with an accuracy of 1 % of the measured value, at the test temperature, 30 min ± 1 min after completing the compression.

8.3.7 Repeat the measurement of the counterforce after different times in accordance with 7.1. Take all measurements at the test temperature.

NOTE 4 After the last measurement at the test temperature, the sample may be allowed to cool down to standard temperature and a further measurement of the counterforce made.

8.4 Method B

8.4.1 Bring the test environment to the test temperature.

8.4.2 When testing in a liquid environment, the test piece and the operating surfaces of the compression device shall be gently lubricated with the test liquid. When testing in a gaseous medium, a thin coating of a lubricant having substantially no action on the rubber shall be applied. A silicone or fluorosilicone fluid, having a kinematic viscosity of about 0,01 m^2/s, and molybdenum disulfide, have been found to be suitable lubricants.

8.4.3 Compress the test piece by (25 ± 2) % at the chosen standard temperature; use a compression of (15 ± 2) % or lower, in steps of 5 %, if a compression of 25 % cannot be obtained. Compress the test piece in a time between 30 s and 2 min. When reached, the final compression shall be fixed and maintained during the entire test period (apart from the further small compression which may be used for measurement of counterforce, as mentioned in 5.2).

8.4.4 Measure the counterforce with an accuracy of 1 % of the measured value, at standard temperature, 30 min ± 1 min after completing the compression.

8.4.5 Immediately after measuring the counterforce, store the compressed test piece in the test environment (see 5.3) at the specified test temperature.

8.4.6 When making measurements of counterforce after the times specified, remove the apparatus from the oven, maintain it at the chosen standard temperature for 2 h, determine the counterforce and then return to the test environment for a further time. It is important that the apparatus and test piece reach thermal equilibrium within 2 h, and forced cooling may be necessary.

8.5 Method C

8.5.1 Bring the test environment to the test temperature.

8.5.2 When testing in a liquid environment, the test piece and the operating surfaces of the compression device shall be gently lubricated with the test liquid. When testing in a gaseous medium, a thin coating of a lubricant having substantially no action on the rubber shall be applied. A silicone or fluorosilicone fluid, having a kinematic viscosity of about 0,01 m^2/s, and molybdenum disulfide, have been found to be suitable lubricants.

8.5.3 Compress the test piece by (25 ± 2) % at the chosen standard temperature; use a compression of (15 ± 2) % or lower, in steps of 5 %, if a compression of 25 % cannot be obtained. Compress the test piece in a time between 30 s and 2 min. When reached, the final compression shall be fixed and maintained during the entire test period (apart from the further small compression which may be used for measurement of counterforce, as mentionned in 5.2).

8.5.4 Within 5 min of completing the compression, immerse the part of the compression device containing the test piece in the test environment at the test temperature.

8.5.5 Two hours after placing the test piece in the test environment, measure the counterforce with an accuracy of 1 % of the measured value at the test temperature.

8.5.6 Repeat the measurement of the counterforce after different times in accordance with 7.1. Take all measurements at the test temperature.

9 Expression of results

The compression stress relaxation, $R(t)$, after a specified duration of test t, expressed as a percentage of the inital counterforce, is given by the equation

$$R(t) = \frac{F_o - F_t}{F_o} \times 100$$

where

F_o is the initial counterforce measured 30 min after completing the compression in methods A and B and 2 h after completing the compression in method C;

F_t is the counterforce measured after the specified duration of test t.

The median value of the results for the test pieces shall be chosen. The individual values for the test pieces shall agree to within 10 % of the median value. If they do not, the test shall be repeated using at least two further test pieces, and the median value of the results from all test pieces shall be chosen and quoted.

Stress relaxation values measured after different times of exposure may be plotted as a function of time on a logarithmic scale to facilitate the interpretation of test data.