INTERNATIONAL STANDARD

ISO 3384

Fourth edition
1999-10-15

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

Reference number
ISO 3384:1999(E)

Contents

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 3.

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 ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

This fourth edition cancels and replaces the third edition (ISO 3384:1991), from which method C has been deleted.

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.

If the lifetime of a material is to be investigated, it can be determined using the air oven ageing test described in ISO 11346.

In addition to the need to specify the temperatures and time intervals in a stress relaxation test, it is 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.

The most important factor in achieving good repeatability and reproducibility when making stress relaxation tests is to keep the temperature and compression constant during all measurements.

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

1 Scope

This International Standard specifies two 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 laboratory 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 normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents listed below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 37:1994, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties.*

ISO 188:1998, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests.*

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

ISO 1817:1999, *Rubber, vulcanized — Determination of the effect of liquids.*

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 4287:1997, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters.*

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

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

ISO 11346:1997, *Rubber, vulcanized or thermoplastic — Estimation of lifetime and maximum temperature of use from an Arrhenius plot.*

3 Term and definition

For the purposes of this International Standard, the following term and definition apply:

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 predetermined test temperature. The decrease in counterforce 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 laboratory temperature. The test pieces are stored at the test temperature.

NOTE 1 The two methods A and B of carrying out the measurement do not give the same values of stress relaxation, and comparison of values obtained from the two 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 may be preferred, and in applications where temperature cycling from normal to an elevated temperature is a problem, method B may be preferred.

NOTE 2 Other methods can be used for specific purposes, such as applying the compression at standard laboratory temperature and making all counterforce measurements at a different temperature.

5 Apparatus

5.1 Compression device, consisting of two parallel, flat, highly polished plates made from chromium-plated or stainless steel or another 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 Ra 0,4 µm (see ISO 4287). When the apparatus is assembled without a 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 and can expand freely laterally.

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 the compression and maintaining it during the whole duration of the test, and it shall be possible to keep the device 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 preferred device is one that monitors the test piece during the whole duration of the test, in which case continuous measurement of the change in counterforce with time is possible. The deformation of the test piece shall be kept within ± 0,01 mm for the duration of the test.

Alternatively, a compression-testing machine can 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. The whole of the force exerted by the test piece as a result of the extra compression shall act on the force-measuring device. It shall also be possible to repeat the compression to within ± 0,01 mm from one measurement to another.

5.3 Test chamber, complying with the requirements of ISO 188:1998, method A.

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 tolerance specified in 7.2. For tests in liquids, the compression device shall be totally immersed in the liquid in a bath, or a closed vessel for volatile or 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 PT 100 element, class A or better. The temperature-sensing element shall be mounted so that it is located not more than 2 mm from a surface of the test piece, in one of the compression plates.

6 Test piece

6.1 Type and preparation of test piece

6.1.1 General

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

NOTE The results obtained from test pieces with different sizes are not comparable.

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.

6.1.3 Ring test pieces

The preferred ring 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 annex B of ISO 37:1994.

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 can be prepared by moulding or from finished articles by cutting and buffing.

Alternatively, an O-ring, size code B0140G as specified in ISO 3601-1:1988 (diameter of the cross-section 2,65 mm and internal diameter 14,0 mm), can be used as the standard test piece.

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

NOTE 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 and 8.4.3 is achieved by careful matching of jig and test piece.

6.2 Measurement of dimensions of test pieces

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

6.3 Number of test pieces

The preferred number of test pieces is three, but for routine and screening tests one or two test pieces are acceptable.

6.4 Time interval between vulcanization and testing

The interval between vulcanization and testing shall be in accordance with ISO 471.

6.5 Conditioning of test pieces

6.5.1 Prior to testing, the test pieces shall undergo first a thermal and then a mechanical conditioning as detailed in 6.5.2 and 6.5.3.

6.5.2 Thermal conditioning shall be carried out by heating the test pieces at 70 °C for 3 h. Following thermal conditioning, the test pieces shall be allowed to stand for a period of not less than 16 h and not more than 48 h at standard laboratory temperature prior to mechanical conditioning or testing.

NOTE Some test samples, especially of thermoplastic elastomers, may contain moulding stresses, and thermal conditioning to relieve these stresses will improve the reproducibility of the results.

6.5.3 Mechanical conditioning shall be carried out at one of the standard laboratory temperatures specified in ISO 471, as follows:

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

Following mechanical conditioning, the test pieces shall be allowed to stand for a period of not less than 16 h and not more than 48 h at standard laboratory temperature prior to testing.

Mechanical conditioning has been found to improve test reproducibility, particularly for compounds containing substantial proportions of filler, but is not always appropriate for finished products and may therefore lead to results that are not typical of service. Such conditioning may be omitted provided thermal conditioning is still undertaken. This omission shall be mentioned in the test report.

7 Duration, temperature and test liquid

7.1 Duration of test

If nothing else is specified, 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 test times are used, a logarithmic time-scale shall be employed.

In method B, when compression is carried out at standard laboratory 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 the list of preferred temperatures in ISO 471.

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

The temperature shall be kept as constant as possible during the test, with a tolerance of ± 1 °C for all temperatures, including standard laboratory temperature.

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. Apply a thin coating of a lubricant having substantially no action on the rubber.

NOTE A silicone or fluoro-silicone fluid (having a kinematic viscosity of about 0,01 m^2/s) and molybdenum disulfide have been found to be suitable lubricants.

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 thermal conditioning and before mechanical conditioning, at the chosen standard laboratory temperature, as specified in 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 after thermal conditioning and before mechanical conditioning, at the chosen standard laboratory temperature, as specified in ISO 4648. Use the average of the measurements to calculate 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, 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 (see 8.1).

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

8.3.4 Place the preheated test piece in the preheated compression device (5.1) or, if the preheating is done in the compression device, place the test piece in the device and then preheat. Compress the test piece by 25 % ± 2 % in the compression device at the test temperature or 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 120 s. When reached, the final compression shall be fixed and maintained during the entire test period (apart from the further small compression which is used for measurement of the counterforce as mentioned in the alternative method in 5.2).

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