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Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression —

Part 2: Testing with temperature cycling

Caoutchouc vulcanisé ou thermoplastique — Détermination de la relaxation de contrainte en compression —

Partie 2: Essais avec cycles de température

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This second edition cancels and replaces the first edition (ISO 3384-2:2012), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the requirement for compression device (5.1) has been harmonized with other International Standards;

— other changes have been made to keep the consistency with ISO 3384-1 throughout the document.

A list of all parts in the ISO 3384 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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”.

Tests in compression are normally made under continuous stress conditions (i.e. the test piece remains strained throughout the test), and are hence a measure of sealing force. Note that the terms continuous and discontinuous used in this standard refer to whether the measure of force is made continuously of at intervals.

Tests to use stress relaxation in tension as a measure of ageing are given in ISO 6914.

The processes responsible for stress relaxation can 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, while at high temperatures and/or long times chemical processes are dominant.

If the life-time of a material is to be investigated, it can be determined using the method 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 can 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 compression constant during all measurements.

The two cycling test methods specified are designed to carry out the following:

— age the test piece by stress relaxation and determine the sealing force at low temperatures (method A);

— introduce thermal stress by stress relaxation and determine the sealing force at low temperatures (method B).

For products used in outdoor applications where the temperature can cycle between a low temperature (e.g. −40 °C) and a high temperature (e.g. 150 °C), it is important to also consider the shrinking of the rubber at low temperatures when assessing performance in the anticipated application and life-time.

For polymers that crystallize at low temperature, the crystallization will add to the shrinking of the rubber. For example, for hoses and seals in automotive applications, the product might work satisfactorily at the normal working temperature, but might leak at a low temperature.

Rubber, vulcanized or thermoplastic — Determination of stress relaxation in compression —

Part 2: Testing with temperature cycling

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document 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 determine the applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document 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 document 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 then undergoes temperature cycling.

Method A: The temperature is cycled at intervals between a high temperature for ageing and a low temperature for checking the sealing force at this low temperature.

Method B: The temperature is cycled continuously between a high temperature and a low temperature to introduce thermal stress in the test piece.

The counterforce is determined by means of a continuous-measurement system.

Two forms of test pieces are specified in this document: cylindrical test pieces and rings. Comparison of results is valid only when made on 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.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 37:2017, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*

ISO 188:2011, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

ISO 23529:2016, *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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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

3.2
thermal stress
mechanical stress induced in a body when some or all of its parts are not free to expand or contract in response to changes in temperature

4 Principle

A test piece of vulcanized or thermoplastic rubber is compressed to a constant deformation at which it is maintained. The decrease in counterforce is then measured.

The temperature is cycled between a high temperature and a low temperature to check the sealing force at this low temperature. The shrinkage of the rubber in going from the high to the low temperature decreases the counterforce.

5 Apparatus

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

The plates shall be:

— sufficiently rigid to ensure that, with a test piece under load, no compression plate bends by more than 0,01 mm;

— of sufficient size to ensure that the whole of the test piece, when compressed between the plates, remains within the area of the plates and can expand freely laterally.

NOTE A surface finish not worse than *Ra* 0,4 µm (see ISO 4287) has been found to be suitable. Such an *Ra* can be obtained by a grinding or polishing operation.

When the apparatus is assembled without a test piece, the gap between the plates shall not vary by more than ±0,1 mm.

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 temperatures. 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 continuous-measurement system monitors the test piece during the whole duration of the test, thus making continuous measurement of the change in counterforce with time possible. The deformation of the test piece shall be kept within ±0,01 mm for the duration of the test. If it is not possible to keep the deformation constant within this tolerance due to the spring effect in load cells, a correction may be done mechanically or mathematically.

5.3 Test environment

5.3.1 For tests in gaseous media, an air oven in accordance with the requirements of ISO 188 shall be used. An oven meeting the requirements specified for one of the ovens used in ISO 188:2011, method A, is recommended.

For cycling the temperature, the oven shall have a cooling and heating capability and be able to change the temperature at a rate of 1,0 °C/min ± 0,5 °C/min.

If the testing is done in nitrogen, oxidative ageing will be eliminated and the result will be due to thermal ageing only. This can be used to simulate conditions where the product is not exposed to air, such as seals used in oil or steam.

5.3.2 For tests in liquids, the compression device shall be totally immersed in a 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.

5.3.3 It is recommended that the air used for air exchange be passed through an air dryer to give it a dew point not higher than −40 °C in order to avoid ice formation which can introduce friction in the measuring system.

5.4 Temperature-measuring equipment, with a sensing element of appropriate precision. The temperature-sensing element shall be installed in such a way that it accurately measures the temperature of the test piece.

NOTE A Pt100 sensor has been found to be suitable for temperature measurement.

6 Calibration

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

7 Test piece

7.1 Type and preparation of test pieces

7.1.1 General

Test pieces shall be prepared either by moulding or by cutting from moulded sheets or products, in accordance with ISO 23529.

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

7.1.2 Cylindrical test pieces

The test piece shall be a cylindrical disc of diameter 13,0 mm ± 0,5 mm and thickness 6,3 mm ± 0,3 mm.

7.1.3 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 A of ISO 37:2017.

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 ISO 3601-1-14 × 2,65-G-N, as specified in ISO 3601-1:2012 (internal diameter 14 mm and diameter of the cross-section 2,65 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.

7.2 Measurement of dimensions of test pieces

The dimensions of test pieces shall be measured as specified in ISO 23529.

7.3 Number of test pieces

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

7.4 Time interval between forming and testing

The interval between forming and testing shall be in accordance with ISO 23529.

7.5 Conditioning of test pieces

7.5.1 Prior to testing, the test pieces shall undergo first thermal and then mechanical conditioning as detailed in 7.5.2 and 7.5.3. When using method A at elevated temperature, the thermal conditioning might not be required, as the preheating of the test piece before compression acts as thermal conditioning.

7.5.2 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, might contain moulding stresses, and thermal conditioning to relieve these stresses might improve the reproducibility of the results.

7.5.3 Mechanical conditioning shall be carried out at one of the standard laboratory temperatures specified in ISO 23529, 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 stress; 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.

