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Rubber, vulcanized or thermoplastic — Determination of ageing characteristics by measurement of stress relaxation in tension

Caoutchouc vulcanisé ou thermoplastique — Détermination des caractéristiques de vieillissement par mesurage de la contrainte de relaxation en traction

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Foreword

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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).

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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 fifth edition cancels and replaces the fourth edition (ISO 6914:2013), which has been technically revised to further align with ISO 3384-1.

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 with time takes place; this behaviour is called “creep”.

The process 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.

Stress relaxation tests in compression are suitable for obtaining a measure of the functional property of sealing force and procedures are given in ISO 3384 (all parts). Stress relaxation tests in tension can be used as a measure of ageing as chemical processes will dominate in the case of a thin test piece exposed to an atmosphere containing oxygen at an elevated temperature for relatively long periods of time.

There are two variants of the technique. Measurements of stress can be made under either

a) continuous strain conditions, or

b) intermittent strain conditions.

In the case of a), continuous strain conditions, the test piece is held in extension throughout the ageing period in the oven. In the case of b), intermittent strain conditions, the test piece is aged in the oven in the unstressed state but, at periodic intervals, it is stretched to a fixed extended length for a short time in order to determine the stress.

NOTE 1 The terms “continuous stress relaxation” and “intermittent stress relaxation” are commonly used to describe the two principal variants of the technique. The latter term, “intermittent stress relaxation”, is a misnomer since no true relaxation of stress occurs and indeed the measured stress can increase with time. For this reason, the use of this term has been avoided in this document although it is fairly well established in the literature.

In a second version of the intermittent test, the test piece is periodically removed from the accelerated ageing atmosphere and the stress is measured under normal laboratory conditions. The advantage of this method is that it does not require the use of special apparatus since a conventional tensile-testing machine can be used for the measurement of stress.

Measurements made in accordance with the methods described in this document provide information about the structural changes that occur in the rubber during ageing, and are complementary: under continuous strain conditions, provided physical relaxation processes are not dominant, the decay of stress provides a measure of the degradative scission reactions in the network. Any new networks formed as a result of crosslinking reactions are considered to be in equilibrium at the test strain with the main network and, therefore, do not impose any new stresses. Under intermittent strain conditions, the decay of stress provides a measure of the net effect of both degradative scission and crosslinking reactions.

NOTE 2 Even under conditions conducive to chemical processes, some physical relaxation can occur. The extent to which it does so will depend on the viscoelastic characteristics of the rubber and on the test conditions and care should be exercised in the interpretation of the results. Physical relaxation is increased by fillers and will be more evident at short times and at lower temperatures. It is often found to be proportional to logarithmic time and is less temperature sensitive than chemical relaxation.

The validity of the methods described in this document depends on the uniformity of degradation in the rubber. For this reason, the thickness of the test pieces used is 1,0 mm to minimize the effect of oxygen diffusion on ageing.

The change in stress can be of direct interest, but the relative resistance of rubbers to ageing will depend on the properties being measured or required by the application. This document should, therefore, be regarded as complementary to ISO 188.

The lifetime of the material, if this is to be investigated, can be determined using the procedures described in ISO 11346.

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

Rubber, vulcanized or thermoplastic — Determination of ageing characteristics by measurement of stress relaxation in tension

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 can 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 describes three methods for the measurement of the change of stress in a test piece at a given elongation for the purpose of determining the ageing characteristics of a rubber.

— Method A is intended for measurement under continuous strain conditions.

— Method B is the preferred method for measurement under intermittent strain conditions.

— Method C is an alternative to method B for measurement under intermittent strain conditions in which the test piece is removed from the ageing environment for measurement of the stress at standard laboratory temperature.

Measurements at a single elevated ageing temperature can be used for quality control purposes as a measure of heat-ageing resistance. Measurements at a number of temperatures can be used for research and development purposes to estimate long-term ageing characteristics in accordance with the procedures described in ISO 11346.

Tests under continuous and intermittent strain conditions measure the results of different processes, and hence do not give the same results. The results of the intermittent methods B and C also cannot be expected to be the same as they use different test conditions.

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

ISO 5893:2019, *Rubber and plastics test equipment — Tensile, flexural and compression types (constant rate of traverse) — Specification*

ISO 18899:2013, *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

No terms and definitions are listed in this document.

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/

4 Principle

A test piece of vulcanized or thermoplastic rubber is elongated to a predetermined tensile strain and the force measured as a function of time in combination with exposure of the test piece to an elevated temperature.

In method A the extension is applied and the force measured over time at the test temperature.

In method B the extension is applied, the force measured and the strain relaxed at the test temperature. This process is repeated at intervals.

In method C the extension is applied, the force measured and the strain relaxed at standard laboratory temperature at intervals between which the test piece is exposed to the test temperature in the relaxed condition.

5 Apparatus

5.1 Stress relaxometer, (for method A or B) consisting of two grips which hold the test piece without slipping at a fixed extended length (to within ± 1 %) together with a means of measuring and recording the force on the test piece.

The grips shall be arranged such that the test piece can be positioned in an oven. The force-measuring system can be, for example, a calibrated spring or electronic load cell, but it shall be accurate and stable to within ± 1 % of the force reading throughout the duration of the test.

For method B, the stress relaxometer shall, in addition, be equipped with a device such that the test piece can be extended and relaxed at intervals. Repeated extension of the test piece shall be constant to within ± 1 % of the elongation.

A tensile machine equipped with a temperature-controlled cabinet can be used as a relaxometer although this is inconvenient for long times and necessitates manual intermittent application of the extension.

5.2 Tensile-testing machine, (for method C) using a constant rate of traverse, operating at 50 mm/min and complying with the requirements specified in ISO 5893:2019, force class 1 (measuring force to within ± 1 % of the measured value).

The machine shall be capable of cycling between fixed strain limits which are accurate to within ± 1 % of the maximum strain. The grips of the tensile-testing machine shall hold the test piece without slippage.

5.3 Oven, complying with the requirements specified for ISO 188:2011, method A (low air speed) or method B (high air speed), for ageing the test piece.

6 Calibration

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

7 Test pieces

7.1 Type and preparation of test pieces

Test pieces shall be parallel-sided strips cut from a sheet. For the tests described in document, it is vital to ensure uniform degradation in the rubber. For this reason, the thickness of standard test pieces shall be (1,0 ± 0,05) mm in order to minimize the effect of oxygen diffusion on ageing.

Samples of uniform thickness of less than 1,0 mm or more than 1,0 mm can be used, but these can give different results.

NOTE For temperatures above 125 °C, reducing the test piece thickness to 0,5 mm is advantageous because of the increased effect of oxygen diffusion at higher temperatures.

Alternatively, product parts or complete products can be used as test pieces taking into account the requirement for thickness.

The other dimensions of the test pieces, i.e. width and length, shall be chosen to suit the sensitivity of the load-measuring device and the precision of the mechanism used for adjusting the strain, in order that the requirements of 5.1 and 5.2 relating to the accuracy of the force and the strain are satisfied.

7.2 Measurement of test piece dimensions

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

7.3 Number of test pieces

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

8 Storage and conditioning

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

Material and test pieces shall be protected from light as much as possible during the interval between vulcanization and testing. They shall not be allowed to come into contact with test sheets and test pieces of a different composition. This is necessary in order to prevent additives which can affect ageing, such as antioxidants, from migrating from one vulcanizate into other vulcanizates.

Test pieces shall be conditioned for a minimum of 3 h at one of the standard laboratory temperatures specified in ISO 23529 immediately before testing.

9 Test conditions

9.1 Duration of test

The duration of test should preferably be chosen from the following series:

1 h, 2 h, 4 h, 8 h, 24 h, 72 h, 168 h, and multiples of 7 d.

For methods A and B, the test period shall be considered to commence when the initial force measurement is made. For method C, the test period shall be considered to be the time in the oven, excluding the time for cooling and the measurement of force.

Alternatively, the test can be stopped when the stress indicator, expressed as the ratio of the force, F_t, at time t to the initial force, F_0 (see Clause 11), reaches a predetermined value (e.g. 0,5).

9.2 Temperature of exposure

The material being tested should preferably be examined at a series of temperatures at intervals of at least 10 °C. If the test pieces are exposed at only one temperature, this shall be chosen from the series of temperatures given in ISO 23529.

The temperature shall be kept as constant as possible during the test, with a tolerance of ± 2 °C for standard laboratory temperature, ± 1 °C for all elevated temperatures up to 100 °C, and ± 2 °C for all elevated temperatures above 100 °C.

It is crucial for the best results that the temperature be kept as stable as possible during the test for two reasons.

— Temperature tolerances in ISO 23529 are ± 1 °C up to and including 100 °C and ± 2 °C for 125 °C up to and including 300 °C. However, studies have shown that a 1 °C change in temperature corresponds to a 10 % difference in ageing time at an Arrhenius factor of 2, or 15 % at a factor of 2,5. This means that two laboratories carrying out ageing at 125 °C can have ageing times which differ by 60 % from each other and still be within the specification. To get accurate results, keep the temperature as accurate as possible by placing a calibrated temperature sensor close to the test pieces and use this to set the oven so that the temperature at this position is correct. Use the correction factor from the calibration certificate to get as close as possible to the true temperature.

— The volume expansion of rubber is 10 to 20 times greater than that of steel, and a temperature variation will cause a variation in the force reading.

As the temperature is increased, the exposure time might need to be reduced. Furthermore, it should be recognized that the greater the disparity between the ageing and the service conditions, the less reliable is the correlation between the ageing and the service life.

10 Procedure

10.1 Method A

Carry out testing in the following manner.

a) Mount the test piece in the preheated grips in the unstrained condition.

b) Position the grips and test piece in the oven preheated to the test temperature.

c) (5 ± 0,5) min after the temperature measured close to the test piece has reached the test temperature, stretch the test piece, in not more than 1 min, to an elongation between 45 % and 55 % and hold it to within 1 % of that elongation. A smaller elongation can be used, for instance in the case of rupture of the test pieces, when (20 ± 2) % is preferred. The initial force, F_0, is taken to be that (5 ± 0,5) min after stretching the test piece.

d) Record the force, F_t on the test piece as a function of time for the duration of the test.

e) At the end of the test, examine the surfaces of the stretched test piece for signs of cracking using a lens with about × 7 magnification. If cracking is found, it shall be reported in the test report.

With certain types of rubber, stress relaxation additional to that caused by oxygen and heat can occur as a result of surface attack by traces of atmospheric ozone. Cracking can invalidate the test and be the cause of variations between measurements.

10.2 Method B

Carry out testing in the following manner.

a) Mount the test piece in preheated grips in the unstrained condition.