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International Standard ISO 3384

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

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

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 3384 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products.*

This second edition cancels and replaces the first edition (ISO 3384-1979), of which it constitutes a minor revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

0 Introduction

When a constant strain is imposed on 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 in time; this behaviour is called creep.

The processes responsible for stress relaxation may be either physical or chemical in nature, and under all normal conditions both processes will occur simultaneously. However, at ambient or low temperatures and/or short times, stress relaxation is dominated by physical processes, whilst at elevated 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 temperature 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.

1 Scope and field of application

This International Standard specifies two methods for determining the decrease in counterforce exerted by a test piece of vulcanized rubber which has been compressed at a constant deformation under specified conditions of time and temperature.

NOTE — 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 must 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.

2 References

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

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

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

ISO 3383, *Rubber — General directions for achieving elevated or sub-normal temperatures for tests.*

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

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

3 Definitions

3.1 stress: The time-dependent force necessary to maintain a constant compressive strain, divided by the original cross-sectional area over which the force is applied.

3.2 stress relaxation: The decrease in stress which has occurred after a specified time-interval, during application of a constant deformation, expressed as a percentage of the stress at the commencement of that time-interval.

4 Principle

4.1 Method A

The test piece is compressed at the test temperature and it is maintained at this temperature throughout the test period, all force measurements being made at the test temperature.

4.2 Method B

The test piece compression and the initial measurement of the counterforce take place at standard temperature; the test piece is then stored in a chamber controlled at the test temperature but it is removed from the chamber for each of the subsequent force measurements, which are made at standard temperature.

5 Apparatus

5.1 Compression device, consisting of two parallel, flat, highly polished stainless steel plates, between the faces of which the test pieces are compressed.

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The finish of the surface of the compression plates shall be not worse than 0,2 µm arithmetical mean deviation from the mean line of the profile (see ISO/R 468). The plates shall be sufficiently rigid to withstand the stress without bending, and of sufficient size to ensure that the whole of the compressed test piece is within the area of the plates.

The compression device shall be connected with suitable equipment for compressing the test piece to the specified compression within 30 s. It shall be capable of setting and maintaining the compression during the whole duration of test and shall be such that it can be kept 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 %. The device may be such as to contain the test pieces during the whole duration of the test, in which case continuous measurements are possible. Alternatively, a testing machine may be used in which the counterforce is measured after prescribed time-intervals on test pieces, compressed in a suitable jig, by applying a slight increase in the compression of the test piece. This additional compression shall be as small as possible and in no case more than a force of 1 N for balance-type machines, and no more than 0,05 mm for stress-strain type machines, applied without overshoot and in a time not greater than 30 s after commencing the additional compression.

5.3 Oven, in accordance with ISO 3383 and provided with temperature control to maintain the specified temperature within the prescribed tolerances. Satisfactory circulation of the air shall be maintained by means of a fan.

5.4 Temperature meter, with for example a thermocouple as the sensing element, mounted in a central position not more than 2 mm distant from the surface in one of the compression plates contacting the test piece.

6 Test piece

6.1 Type and preparation of test piece

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

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

Cutting is carried out by means of a sharp, rotating, circular die or revolving knife, lubricated with soapy water, and brought carefully into contact with the rubber. Alternatively, the die or knife is kept stationary and the rubber rotated against it.

The rubber is mounted on suitable backing material and the cutting pressure is kept small enough to avoid "cupping" of the cut surface.

6.2 Number of test pieces

At least three test pieces shall be used for each test.

6.3 Time-interval between vulcanization and testing

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

6.4 Conditioning of test pieces

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 6.4.1. Alternatively, thermal conditioning only shall be carried out as specified in 6.4.2.

6.4.1 Mechanical conditioning shall be applied at one of the standard laboratory 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.

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

6.4.2 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 laboratory temperatures for a period of 3 h immediately before testing.

7 Procedure

7.1 Carefully clean the operating surfaces of the compression device. The test piece surface shall be free from mould release agent or dusting powder. 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.

7.2 Measure the thickness of each test piece at the central portion with an accuracy of 0,01 mm after conditioning at the chosen standard laboratory temperature (see 6.4). The thickness shall be determined according to ISO 4648, method A1.

7.3 Method A

7.3.1 Preheat the compression device to the test temperature.

7.3.2 Preheat the test piece to the test temperature in accordance with ISO 3383. A preheating period of 30 min is recommended.

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7.3.3 Compress the preheated test piece by (25 ± 2) % in the compression device (5.1) at the test temperature; use a compression of (15 ± 2) % if a compression of 25 % cannot be obtained. Apply the compression within 30 s. 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, mentioned in 5.2).

7.3.4 $30\ ^{+2}_{0}$ min after applying the compression, measure the counterforce with an accuracy of ± 1 %, still at the test temperature.

7.3.5 Repeat the measurement of the counterforce after different times, according to 8.1. Take all measurements at the test temperature.

7.4 Method B

7.4.1 Compress the test piece by (25 ± 2) % at the chosen standard laboratory temperature (see 6.4); a compression of (15 ± 2) % may be used when a compression of 25 % cannot be obtained. Apply the compression within 30 s. 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, mentioned in 5.2).

7.4.2 $30\ ^{+2}_{0}$ min after applying the compression, measure the counterforce with an accuracy of ± 1 %, still at the standard laboratory temperature.

7.4.3 Store the compressed test piece in the oven (5.3) at the specified elevated temperature.

7.4.4 When making measurements of counterforce after the times specified, remove the apparatus from the oven, maintain it at the chosen standard laboratory 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.

7.5 Carry out temperature control during the test period using the sensing element mounted in one of the compression plates as prescribed in 5.4.

8 Duration and temperature of test

8.1 Duration of test

The duration of test shall be $168\ ^{0}_{-2}$ h. If intermediate times are used, $24\ ^{0}_{-0,5}$ h and $72\ ^{0}_{-1}$ h are preferred. The test period commences after the initial compression. If longer testing times are needed, a logarithmic time-scale shall be used.

In method B, the 2 h required after each time-interval, to attain standard laboratory temperature, shall not be included in the duration of test.

8.2 Temperature of test

The temperature of test shall be chosen from the following list of preferred temperatures.

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

Unless for technical reasons another temperature is required, the ambient and elevated test temperatures shall be 23 °C, 27 °C and 100 °C, respectively.

9 Expression of results

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

$$R(t) = \frac{F_0 - F_t}{F_0} \times 100$$

where

F_0 is the initial counterforce measured 30 min after compression of the test piece;

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

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

NOTE — For some applications, it may be more useful to calculate compression stress ratio values, i.e. F_t/F_0, at various times after the compression is applied, rather than stress relaxation values. The compression stress ratio values may be presented graphically as a function of time. The use of a logarithmic scale for the time-axis may facilitate extrapolation of the test data.

* Standard laboratory temperatures.

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10 Test report

The test report shall include the following information:

a) Sample details:

1) a full description of the sample and its origin;

2) compound details, cure time and temperature and date of cure, where appropriate;

3) method of preparation of test pieces from sample.

b) Test method and test details:

1) the number of this International Standard;

2) the method used, A or B;

3) the standard laboratory temperature;

4) the duration and temperature of test;

5) the type and dimensions of test pieces;

6) nature of lubricant, if used;

7) a description of the principles of the testing device (oven included);

8) the method used for measuring the counterforce;

9) any non-standardized procedures, for example, mechanical conditioning.

c) Test results:

1) the number of test pieces tested;

2) the median value of the test results, expressed according to clause 9.

d) Date of test.

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