INTERNATIONAL STANDARD

ISO 12493

Second edition
2017-07

Rubber, vulcanized — Determination of stress in tension upon heating

Caoutchouc vulcanisé — Détermination de la contrainte en traction lors du chauffage

Reference number
ISO 12493:2017(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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 on 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 the following URL: 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 12493:2011), of which it constitutes a minor revision to update the normative references in Clause 2.

Introduction

Vulcanized rubber held under a constant stress will contract as the test temperature is raised, while a test piece held under a constant strain will develop an increased stress. These are features of the Gough-Joule effect in rubber and, unless they are taken into account at the design stage, any resulting changes in forces and dimensions can affect the performance of some products, such as rotary seals, used at high temperatures and high strains (see Reference [1]).

This document describes a test method for the determination of the change in tensile stress that results from an increase in test temperature.

Rubber, vulcanized — Determination of stress in tension upon heating

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 ensure compliance with any national regulatory conditions.

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 a method for measuring the stress in tension which is developed in vulcanized rubber when it is heated (thermal stress). The thermal stress is measured for various pre-strain and temperature conditions as a function of time.

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 5893, *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

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 http://www.iso.org/obp

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

3.1
thermal stress
σ_T
force per initial unit area which is developed in the test piece upon heating

Note 1 to entry: It is expressed in N/m^2 or Pa.

3.2
maximum thermal stress
max. σ_T
peak value of the thermal stress recorded during the test

3.3
thermal stress after a specified time
$\sigma_{T,t}$
stress induced in the test piece upon heating for a specified time t

3.4
pre-strain
elongation to which the test piece is subjected at the beginning of the test

Note 1 to entry: It is expressed as:

$$\text{pre-strain} = \frac{l_f - l_i}{l_i}$$

where

l_i is the initial length;

l_f is the length after elongation.

3.5
pre-stress
force per initial unit area which results from the pre-strain

Note 1 to entry: It is expressed in N/m^2 or Pa.

4 Principle

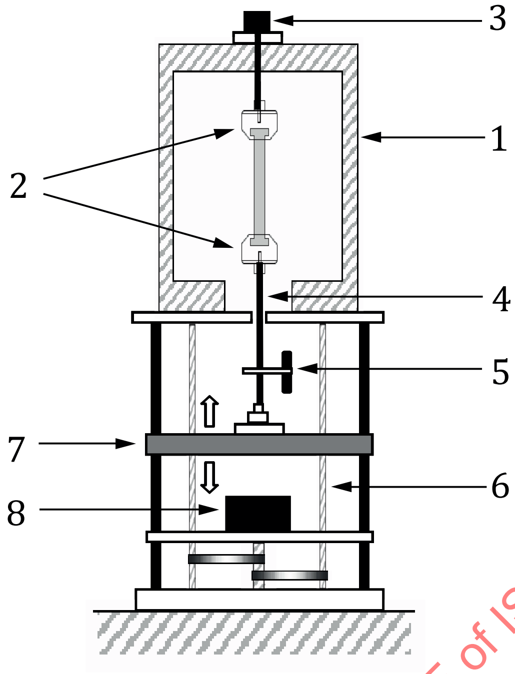
A test piece is held at a constant pre-strain in a tensile mode at standard laboratory temperature. When the pre-stress resulting from the given pre-strain has reached an apparent equilibrium value, the temperature of the test piece is increased. The thermal stress developed at the elevated temperature is measured for various pre-strain conditions as a function of time.

5 Apparatus

5.1 Thermal-stress testing machine

An example of a test machine for measuring the thermal stress developed in rubbery materials when heated is shown in Figure 1. Two clamps hold the test piece in a temperature-controlled chamber, with the upper clamp connected to a load cell and the bottom clamp connected to a crosshead. The crosshead is moved using a screw driven by a motor to impose a pre-strain on the test piece. The thermal stress developed when the temperature is raised is transmitted to the load cell and the output is recorded to give the variation in stress as a function of time.

The test machine shall comply with ISO 5893 with force measurement to class 1 and the machine shall be capable of setting the pre-strain to within ± 0,1 at a speed of (20 ± 2,5) mm/min.



Key

1 temperature-controlled chamber
2 clamps
3 load cell
4 rod
5 linear variable differential transformer
6 screw
7 crosshead
8 motor

Figure 1 — Example of thermal-stress testing machine

5.2 Temperature-controlled chamber

The temperature-controlled chamber shall be capable of raising the temperature at a rate of at least 30 °C/min and maintaining the test piece at the required temperature as specified in ISO 23529. A suitable volume for the chamber is 3 l to 5 l. A temperature-sensing device shall be located within the chamber near the test piece.

5.3 Thickness- and width-measuring devices

Instruments for measuring the thickness and width of the test piece shall be in accordance with ISO 23529.

6 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in Annex A.

7 Test piece

7.1 Dimensions

The test piece shall be prepared by cutting from moulded flat sheet and shall have the shape and dimensions shown in Figure 2. In addition, the thickness shall be (2 ± 0,2) mm. The test piece shall have a smooth surface and be free from irregularities.

Dimensions in millimetres

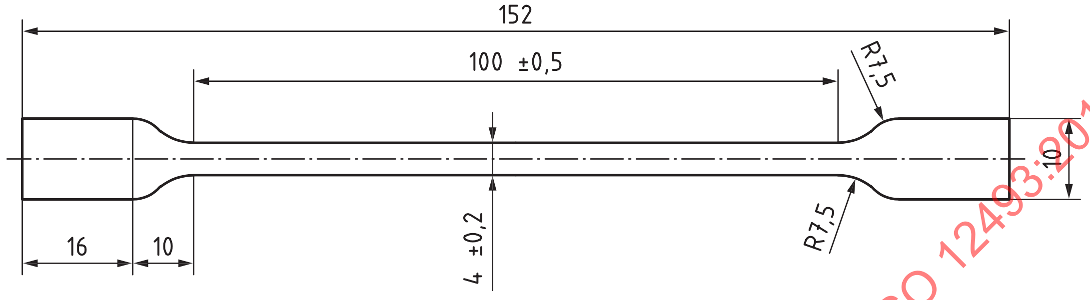


Figure 2 — Test piece for thermal-stress measurements

7.2 Number of test pieces

At least three test pieces shall be used for each set of test conditions.

7.3 Time lapse between moulding and testing

Unless otherwise specified, the following requirements shall be observed (see ISO 23529):

— For all test purposes, the minimum time between moulding and testing shall be 16 h.

— For non-product tests, the maximum time between vulcanization and testing shall be four weeks and, for evaluations intended to be comparable, the tests, as far as possible, shall be carried out after the same time interval.

— For product tests, whenever possible, the time between vulcanization and testing shall not exceed three months. In other cases, tests shall be made within two months of the date of receipt of the product by the customer.

7.4 Conditioning

Test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

Test pieces shall be conditioned at a standard laboratory temperature for at least 3 h immediately before being measured and tested.

If the test is to be carried out at a starting temperature other than a standard laboratory temperature, the test pieces shall be conditioned at the test temperature immediately prior to testing for a period sufficient to ensure that they have reached the test temperature (see ISO 23529).

8 Test conditions

8.1 Temperature

The elevated temperatures to be used shall be selected from those specified in ISO 23529 unless otherwise necessary for technical reasons. Recommended test temperatures are 60 °C, 100 °C and 140 °C.

8.2 Pre-strain

A minimum of three pre-strains at each temperature shall be selected. Recommended pre-strains are 0,2, 0,4 and 0,6.

9 Procedure

Mount a test piece in the clamps at a standard laboratory temperature and elongate it to the required pre-strain by movement of the crosshead at a speed of (20 ± 2,5) mm/min, as shown in Figure 3. Hold the test piece at the constant pre-strain until the pre-stress resulting from the pre-strain reaches an apparent equilibrium value. This will be after about 30 min. Then, reset the load cell to zero and increase the temperature to the required test temperature. Start recording the thermal stress immediately after the heating begins (designated as zero time). Monitor the development and decay of the thermal stress as a function of time. The minimum duration of the test shall be 30 min.

Unless otherwise specified, carry out the test to obtain results at a minimum of three temperatures, with three pre-strains at each temperature.

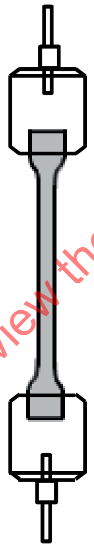
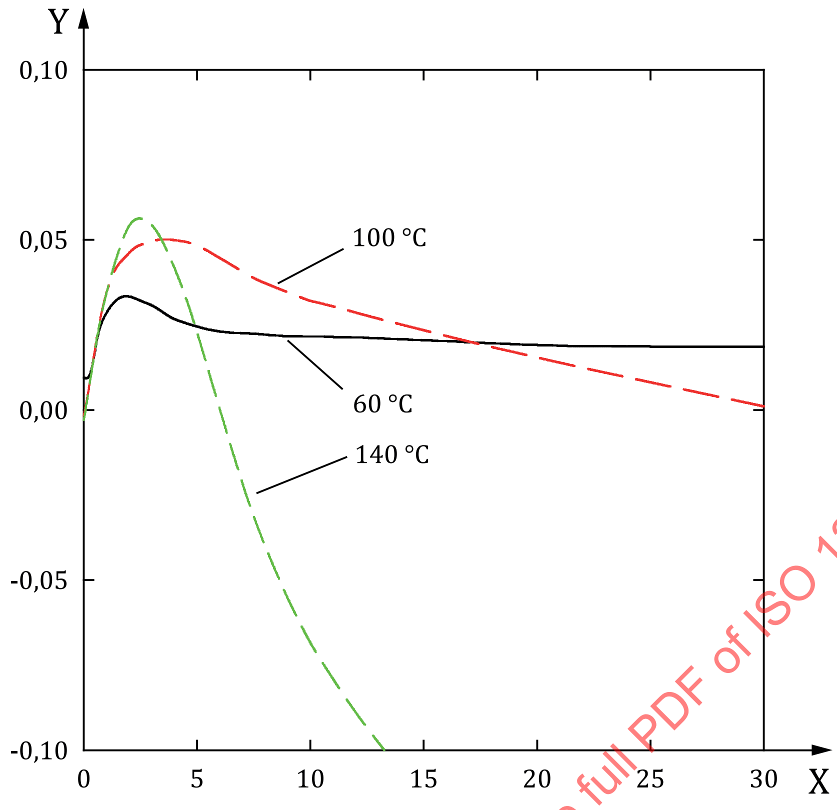


Figure 3 — Schematic diagram of a test piece with clamps at a constant pre-strain

10 Expression of results

Plot the thermal stress in the test piece as a function of time. From the graph, obtain the maximum thermal stress, the time to reach the maximum thermal stress, and the thermal stress at a specified time, which shall be taken as 20 min unless otherwise specified.

An example of the thermal stress of an unfilled natural rubber (NR) vulcanizate at a pre-strain of 0,4 is shown in Figure 4 as a function of time at three different temperatures.



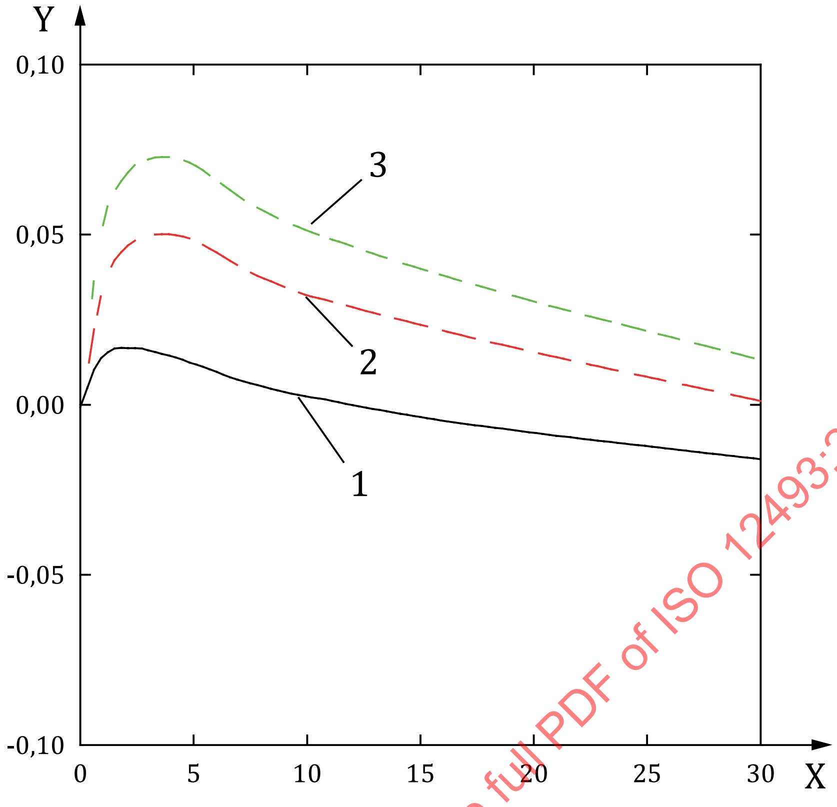
Key

X t, min

Y σ_T, MPa

Figure 4 — Thermal stress at various temperatures of an unfilled NR vulcanizate at a pre-strain of 0,4

Another example of the thermal stress of an unfilled NR vulcanizate at a test temperature of 100 °C is shown in Figure 5 as a function of time at three different pre-strains.



Key

X t, min

Y σ_T, MPa

1 pre-strain 0,2

2 pre-strain 0,4

3 pre-strain 0,6

Figure 5 — Thermal stress at various pre-strains of an unfilled NR vulcanizate at a test temperature of 100 °C

11 Precision

No precision data are currently available for this method.

12 Test report

The test report shall include the following information:

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

b) a full reference to the test method used, i.e. the number of this document (ISO 12493);

c) test details:

1) the standard laboratory temperature used,

2) the time and temperature of conditioning prior to the test,

3) the test temperatures used,

4) the pre-strains used,

5) details of any procedures not specified in this document;

d) test results:

1) the number of test pieces used,

2) the graph(s) plotted of thermal stress against time,

3) the mean values of the individual results from the graph(s) — maximum thermal stress (max. σ_T), time to maximum thermal stress, and thermal stress after a specified time ($\sigma_{T,\ t}$);

e) the date of the test.