

BS ISO 12493:2011



BSI Standards Publication

Rubber, vulcanized — Determination of stress in tension upon heating

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National foreword

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**Rubber, vulcanized — Determination of
stress in tension upon heating**

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





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Foreword

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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.

ISO 12493 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

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] in the Bibliography).

This International Standard 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

1 Scope

This International Standard 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 referenced documents are indispensable for the application 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 18899:2004, *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.

3.1

thermal stress

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

NOTE It is expressed in N/m² 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 It is expressed as 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 It is expressed in N/m² or Pa.

4 Principle

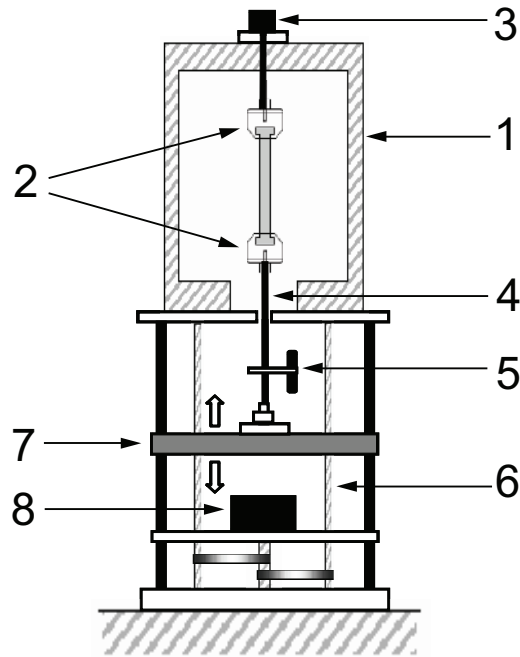
A test piece is held at a constant pre-strain in a tensile mode at standard laboratory temperature. When the pre-stress resulting from the given pre-strain has reached an apparent equilibrium value, the temperature of the test piece is increased. The thermal stress developed at the elevated temperature 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 $\pm 0,1$ at a speed of $(20 \pm 2,5)$ 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 litres to 5 litres. 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 $(2 \pm 0,2)$ mm. The test piece shall have a smooth surface and be free from irregularities.

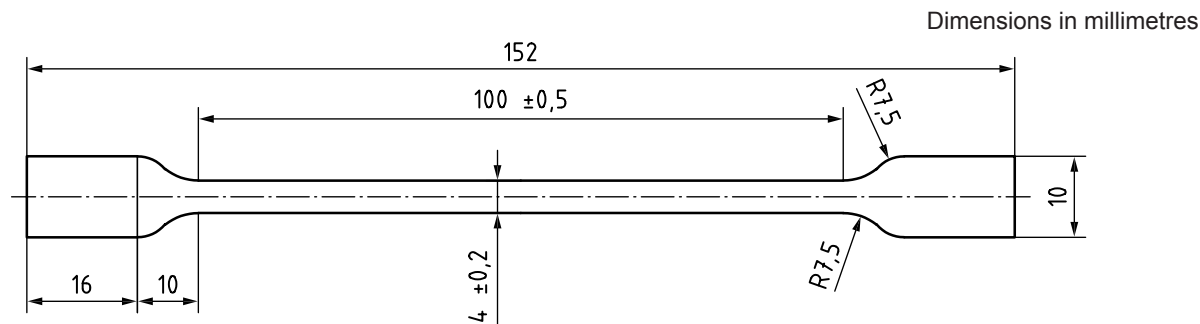


Figure 2 — Test piece for thermal-stress measurements

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 $(20 \pm 2,5)$ 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.

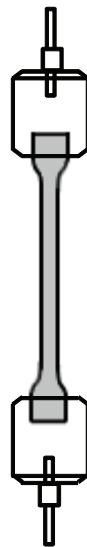


Figure 3 — Schematic diagram of a test piece with clamps at a constant pre-strain

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.

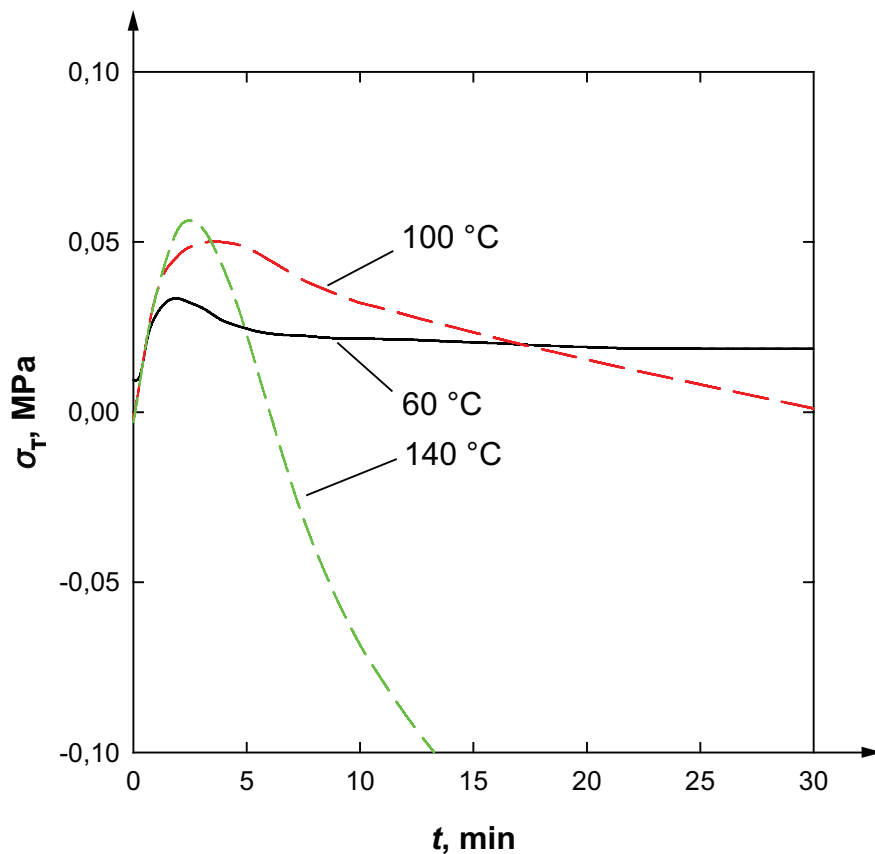
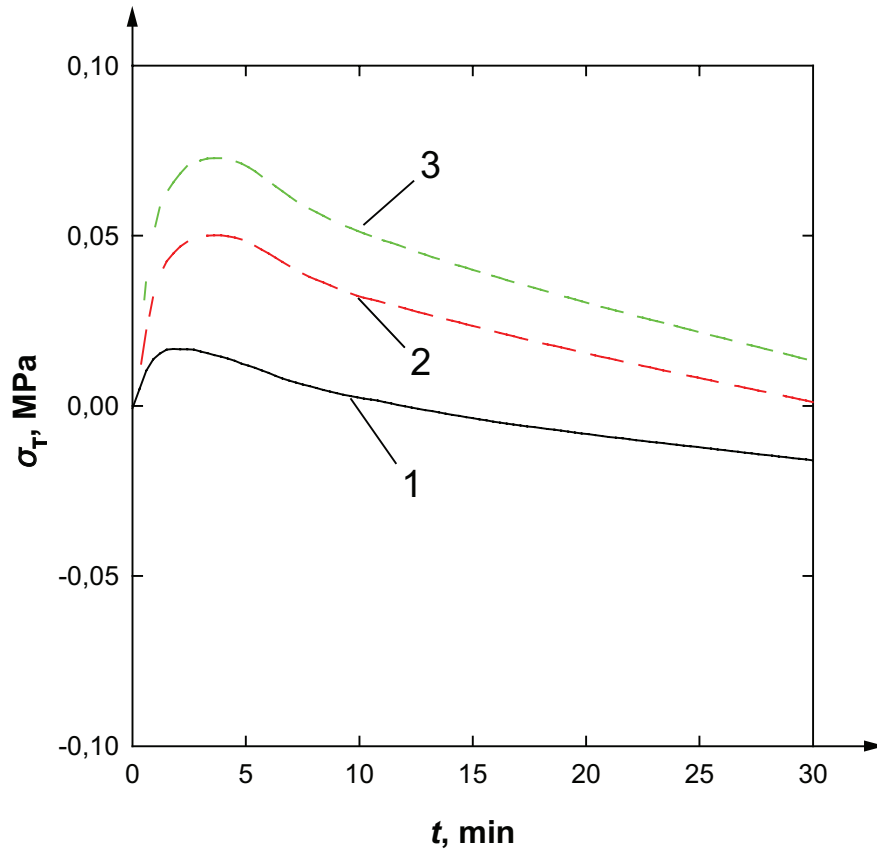


Figure 4 — Thermal stress at various temperatures of an unfilled NR vulcanizate at a pre-strain of 0,4

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

- 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 International Standard;
- 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 International Standard;
- 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 (σ_T, t);
- e) the date of the test.

Annex A (normative)

Calibration schedule

A.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded in any calibration report or certificate. It shall be reported whether calibration is carried out in the “as-received” condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

A.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this International Standard. However, the frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table A.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that in ISO 18899 is available, it shall be used in preference).

The code-letter S used in the calibration schedule indicates the standard interval as given in ISO 18899.

Table A.1 — Calibration frequency schedule

Parameter	Requirement	Subclause in ISO 18899:2004	Verification frequency guide	Notes
Test machine	Complying with ISO 5893	—	S	
Force measurement	Class 1 of ISO 5893	21.1	S	
Pre-strain	±0,1	15.2	S	
Crosshead speed	(20 ± 2,5) mm/min	23.4	S	
Rate of increase of temperature in temperature-controlled cabinet	30 °C/min minimum	23.6	S	

In addition to the items listed in the table, use of the following is implied, all of which need calibrating in accordance with ISO 18899:

- timer;
- thermometers for monitoring the conditioning and test temperatures;

- instruments for determining the dimensions of the test piece.

Bibliography

- [1] HONG, C.K., PARK, S., and KAANG, S., A test method for measuring the dimensional stability of elastomeric materials upon heating, *Polymer Testing*, **27**(2), pp. 146-152 (2008)

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