INTERNATIONAL STANDARD

ISO 12493

Third edition
2023-01

Rubber, vulcanized or thermoplastic — Determination of stress in tension under non-isothermal conditions

Caoutchouc vulcanisé ou thermoplastique — Détermination de la contrainte en traction dans des conditions non isothermes

Reference number
ISO 12493:2023(E)

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

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

This third edition cancels and replaces the second edition (ISO 12493:2017), which has been technically revised.

The main changes are as follows:

— another method (method B) for measuring tensile stress under non-isothermal conditions has been added. In this method, the change of stress is measured at a given strain and under variation of temperature at a given heating rate as a function of temperature.

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Introduction

Usually, stress relaxation tests (see ISO 6914) are performed at constant temperature conditions because temperature has a strong impact on the relaxation time constants of rubber materials. Depending on the respective relaxation time constant or, more realistic, relaxation time spectrum, those measurements are more or less time consuming and can require testing times of several days, weeks or even months. Accelerated stress relaxation tests can be performed under non-isothermal conditions, if the temperature is increased at a certain constant heating rate, because relaxation processes are thermally activated and therefore occur faster at higher temperatures. Thus, the entire stress relaxation behaviour of the material can be scanned within a short period of time, typically a few hours. This method is designated as the temperature scanning stress relaxation (TSSR) test method and offers the ability to characterize rubber — vulcanized or thermoplastic — by short-term measurements. TSSR tests cannot replace conventional isothermal stress relaxation measurements, but are considered as a comparative test method, e.g. for the purpose of material pre-selection or in order to determine the state of crosslink density — which is an important reason for ageing phenomena — of a sample.

During non-isothermal testing, the material undergoes not only stress relaxation, but additional phenomena occur which need to be considered by adequate corrections. Most important in this case is an increase of retractive forces due to the Gough-Joule effect and thermal expansion of the sample. Whereas the latter can be numerically compensated by considering a proper value of the coefficient of thermal expansion (CTE), the increase of retractive forces offer the opportunity to calculate the crosslink density of the material, based on fundamental theory of rubber elasticity. Furthermore, an enhanced sensitivity of the test, with respect to specific relaxation processes is achieved by determination of the first derivative of the measured stress–temperature curve. Similar to stress relaxation measurements in the time domain, the latter can be used to calculate a corresponding relaxation temperature spectrum, instead of a relaxation time spectrum.

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Rubber, vulcanized or thermoplastic — Determination of stress in tension under non-isothermal conditions

WARNING 1 — Users of 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 users to establish appropriate safety and health practices and 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 two methods for measuring stress in tension under non-isothermal conditions.

— Method A: The thermal stress is measured for various pre-strain and temperature conditions as a function of time.

— Method B: The change of stress is measured in a test piece at a given strain and under variation of temperature at a given heating rate as a function of temperature. In this way, the determination of the thermal-mechanical behaviour of a rubber can be accelerated, e.g. for the purpose of comparative testing of aging or estimating the upper limit of the operating temperature.

The measurement device, which is equipped with a suitable heating chamber, is used to record the stress as a function of time or temperature until the sample breaks or the stress has approached zero or for a certain 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 188:2011, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

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 terminology databases for use in standardization at the following addresses:

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

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

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3.1
thermal stress
σ_T
force per initial unit area that 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
$\sigma_{T,max}$
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
strain to which the test piece is subjected at the beginning of the test

Note 1 to entry: It is expressed as:

$$\frac{l_f - l_i}{l_i} * 100$$

where

l_i is the initial length;

l_f is the length after strain.

3.5
pre-stress
force per initial unit area that 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 a constant 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. In method A, the test temperature is reached as quickly as possible, in method B, heating is carried out at a constant rate. The thermal stress developed at the elevated temperature is measured.

5 Apparatus

5.1 Test machine

5.1.1 General

The setup of the measuring devices can be done in different ways. Two possibilities are described below.

5.1.2 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

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