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Plastics — Thermomechanical analysis (TMA) —

Part 2:
Determination of coefficient of linear thermal expansion and glass transition temperature

Plastiques — Analyse thermomécanique (TMA) —

Partie 2: Détermination du coefficient de dilatation thermique linéique et de la température de transition vitreuse

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Contents

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11359-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties.*

ISO 11359 consists of the following parts, under the general title *Plastics — Thermomechanical analysis:*

— *Part 1: General principles*

— *Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*

— *Part 3: Determination of penetration temperature*

Annex A of this part of ISO 11359 is given for information only.

Plastics — Thermomechanical analysis (TMA) —

Part 2:
Determination of coefficient of linear thermal expansion and glass transition temperature

1 Scope

This part of ISO 11359 specifies a test method, using thermodilatometry, for the determination of the coefficient of linear thermal expansion of plastics in a solid state by thermomechanical analysis (TMA). This part of ISO 11359 also specifies the determination of the glass transition temperature using TMA.

NOTE The coefficient of linear thermal expansion can be measured using various types of thermodilatometry apparatus. This part of ISO 11359 concerns only TMA apparatus.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 11359. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 11359 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 291, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 11359-1, *Plastics — Thermomechanical analysis (TMA) — Part 1: General principles.*

3 Terms and definitions

For the purposes of this part of ISO 11359, the terms and definitions given in ISO 11359-1 apply, plus the following:

3.1
thermal expansion
increase in dimensions of a specimen as a function of temperature, measured by thermodilatometry

3.2
coefficient of linear thermal expansion
reversible increase in length of a material per unit length per degree change in temperature

NOTE Two different coefficients of thermal expansion can be determined: the differential coefficient of linear thermal expansion and the mean coefficient of linear thermal expansion.

3.2.1
differential coefficient of linear thermal expansion
α
coefficient of expansion for any of the three dimensions at temperature T and at constant pressure p given, in reciprocal kelvins, by the following equation:

$$\alpha = \frac{(\mathrm{d}L)_p}{(\mathrm{d}T)_p} \times \frac{1}{L_0} = \frac{(\mathrm{d}L/\mathrm{d}t)_p}{(\mathrm{d}T/\mathrm{d}t)_p} \times \frac{1}{L_0} \qquad (1)$$

where

L_0 is the reference length at room temperature T_0, in the axis of measurement;

L is the length at temperature T, in the axis of measurement;

$\mathrm{d}L$ is the change in length over the time interval $\mathrm{d}t$ at constant pressure p;

$\mathrm{d}T$ is the change in temperature over the time interval $\mathrm{d}t$ at constant pressure p.

3.2.2
mean coefficient of linear thermal expansion
$\overline{\alpha}$
coefficient of expansion for any of the three dimensions at constant pressure, given, in reciprocal kelvins, by the following equation:

$$\overline{\alpha} = \frac{\Delta L}{\Delta T} \times \frac{1}{L_0} \qquad (2)$$

where

ΔL is the change in length of the test specimen between two temperatures T_1 and T_2;

L_0 is the reference length of the test specimen at room temperature in the axis of measurement;

ΔT is the change in temperature, equal to $T_2 - T_1$.

The determination is made over a temperature interval ΔT between T_1 and T_2. The representative temperature is given by

$$T(\text{representative}) = \frac{T_1 + T_2}{2}$$

NOTE By replacing the term “length” by “volume” in equations (1) and (2), the coefficient of volumetric thermal expansion can be obtained.

3.3
glass transition
reversible change in an amorphous polymer or in the amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery state to (or from) a hard and relatively brittle one

3.4
glass transition temperature
T_g
approximate midpoint of the temperature range over which the glass transition takes place

The glass transition temperature obtained by thermodilatometry is defined as the point of intersection of the tangents to the length versus temperature curve before and after the glass transition (see Figure 3).

4 Principle

The change in a dimension of a test specimen is measured as a function of temperature using a TMA apparatus to generate a TMA curve from which the coefficient of linear thermal expansion can be calculated.

5 Apparatus

The components of the TMA apparatus used for this part of ISO 11359 shall be as specified in ISO 11359-1 and shall also be capable of:

a) operating in a compression mode or a tension mode or both;

b) maintaining the specimen under a controlled atmosphere in accordance with ISO 291.

NOTE 1 Measurements on specimens of film or fibre are made in the tension mode.

NOTE 2 It is preferable to use an atmosphere of dry air or an inert gas such as nitrogen.

6 Test specimens

6.1 Preparation

Prepare test specimens in accordance with ISO 11359-1, clause 7.

The standard test specimen is a rectangular specimen 5 mm to 10 mm in length and about 5 mm in width. However, specimens of other dimensions may be used by agreement between the interested parties. The ends of the test specimen shall be parallel. Record, if applicable, the orientation of the specimen with respect to the direction of production, i.e. machine direction, transverse direction or other.

Refer to the relevant material standards for the number of test specimens, but prepare and test at least three specimens from each sample.

6.2 Conditioning

Refer to the relevant material standards for the conditioning of specimens before measurement.

NOTE 1 In order to eliminate any thermal-memory effects in the specimen, it is preferable that each specimen be heated from the minimum measurement temperature (at least 50 °C below T_g) to the maximum temperature (at least 50 °C above T_g), then held at this latter temperature for 5 min. Subsequently, cool the specimen to the minimum temperature at the same rate as that which will be used for the actual determination.

NOTE 2 Heating the test specimen to temperatures of 50 °C above T_g may result in changes in molecular orientation and/or blend morphology, thereby changing the coefficient of linear thermal expansion in certain directions.

7 Procedure

7.1 Calibration of apparatus

Calibrate the apparatus in accordance with ISO 11359-1. After cleaning the surfaces of the specimen, probe and sample holder, place the specimen on the sample holder with the probe as close as possible.

7.2 Determination

Set the unloaded probe on the upper surface of the specimen. Apply a load of preferably 4,0 kPa ± 0,1 kPa. Provided the effect on the measured value is small, other loads may be used.

When specimens made of film, fibre or soft material are tested, carry out the determination in the tension mode with both ends of the specimen gripped.

Maintain a constant gas flow, preferably of dry air, around the specimen within a flowrate range of 50 ml/min to 100 ml/min. However, other atmospheres may be used by agreement between the interested parties.

Increase the temperature of the specimen at a constant rate of not more than 5 °C/min.

Record the TMA curve for the test specimen, i.e. the change in length with increasing temperature.

NOTE 1 It is preferable with some samples that specimens taken in different directions from the sample be investigated.

Measure, under the same conditions, the change in length of a reference specimen having a known mean coefficient of linear thermal expansion and about the same length as the test specimen.

NOTE 2 This is not necessary when the apparatus used measures the difference in length between the test specimen and a reference specimen directly.

8 Expression of results

8.1 Method of calculation

8.1.1 Coefficient of linear thermal expansion α

The coefficient of linear thermal expansion α, in reciprocal kelvins (K^{-1}), at temperature T is obtained from the TMA curve using the following equation (see Figure 1):

$$\alpha = \frac{dL}{dT} \times \frac{1}{L_0}$$

where

L_0 is the length of the specimen, in micrometres, at room temperature;

L is the length of the specimen, in micrometres, at temperature T;

T is in kelvins.

Calculate the value of α to the nearest 10^{-7} K^{-1} for each specimen. Calculate the mean of these individual values and round the mean to the nearest 10^{-6} K^{-1}.

In the case of a test specimen exhibiting a glass transition, calculate the coefficient before and after the glass transition.

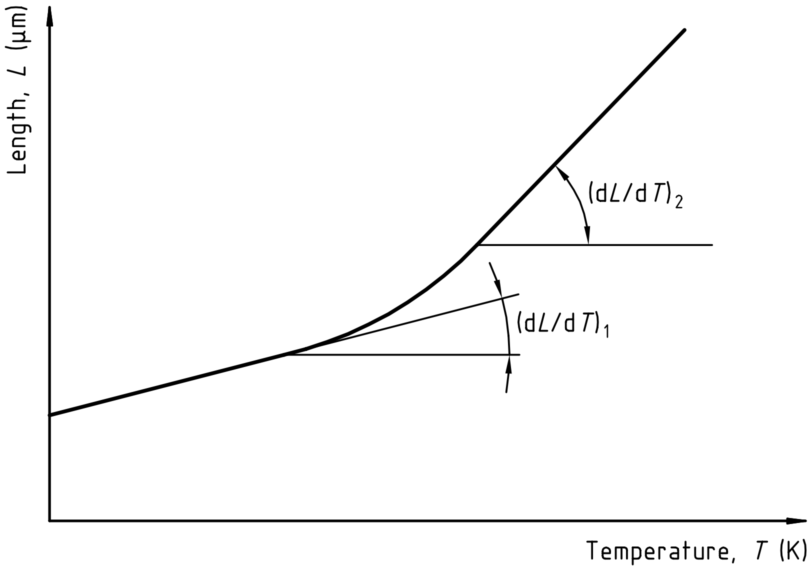

Figure 1 — Determination of coefficient of linear thermal expansion α

8.1.2 Mean coefficient of linear thermal expansion $\overline{\alpha}$

8.1.2.1 Method A: Determination without reference specimen

The mean coefficient of linear thermal expansion $\overline{\alpha}$, in reciprocal kelvins (K^{-1}), between two temperatures T_1 and T_2 is obtained from the TMA curve using the following equation (see Figure 2):

$$\overline{\alpha} = \frac{\Delta L}{\Delta T} \times \frac{1}{L_0}$$

where

L_0 is the length of the specimen, in micrometres, at room temperature;

ΔL is the difference in length, in micrometres;

ΔT (= $T_2 - T_1$) is the temperature difference, in kelvins.

Select two temperatures and calculate the difference in temperature ΔT.

Determine the corresponding change in length ΔL from the TMA curve.

Calculate the value of $\overline{\alpha}$ to the nearest 10^{-7} K^{-1} for each specimen. Calculate the mean of these individual values and round the mean to the nearest 10^{-6} K^{-1}.

In the case of a test specimen exhibiting a glass transition, calculate the coefficient before and after the glass transition.