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Plastics — Determination of dynamic mechanical properties —

Part 11: Glass transition temperature

Plastiques — Détermination des propriétés mécaniques dynamiques —

Partie 11: Température de transition vitreuse

Reference number
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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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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 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 6721-11:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

— the scope has been revised to specify suitable materials more accurately;

— definitions of specific points in DMA curves have been extended;

— reference to quality assurance purposes have been deleted;

— several methods have been introduced for evaluation of the glass transition temperature;

— the procedure for determination of heat dependent results has been revised;

— curves of storage modulus, loss modulus and loss factor have been added to the test report;

— the temperature calibration procedure has been revised;

— additional temperature reference specimen for different loading modes has been introduced.

A list of all parts in the ISO 6721 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at https://www.iso.org/members.html.

Introduction

This document covers the use of dynamic mechanical analysis (DMA) procedures, in the temperature scanning mode, to determine a value for the glass transition temperature of plastics. It provides an alternative procedure to the use of differential scanning calorimetry (DSC) (see ISO 11357-2)[1] for this measurement.

DMA is used to determine the variation of the storage modulus, loss modulus and loss factor as a function of temperature and frequency. From these data, a value for the glass transition temperature is determined. Many types of commercial equipment are available that use this technique, and, in principle, it applies to all the loading modes described in ISO 6721-1.

The procedures minimize errors due to thermal lag of the specimen, which varies with the heating rate used, through assuming the specimen temperature is given by the measured oven temperature[2]. This eliminates the need for the temperature of the specimen to be measured directly by, for example, a thermocouple embedded in the specimen.

Plastics — Determination of dynamic mechanical properties —

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WARNING — The use of this document may involve hazardous materials, operations and equipment. The document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and to determine the applicability of any other restrictions prior to its use.

1 Scope

This document specifies methods for determining a value of the glass transition temperature (T_g) from the dynamic mechanical properties measured during a linear temperature scan under heating conditions. The glass transition temperature is an indicator of the transition from a hard and relatively brittle glassy state to a rubbery or viscous liquid state in an amorphous polymer or in amorphous regions of a partially crystalline polymer.

Usually referred to as dynamic mechanical analysis (DMA), the methods and their associated procedures can be applied to unreinforced and filled polymers, foams, rubbers, adhesives and fibre-reinforced plastics/composites. The methods are limited to materials that are inherently stable above T_g, i.e. amorphous materials that transform into a rubbery state or partially crystalline materials that keep their shape due to crystallinity.

Different modes (e.g. flexure, torsion, shear, compression, tension) of dynamic mechanical analysis can be applied, as appropriate, to the form of the source material.

Measured T_g values using instrumentation can vary as a result of material characteristics and/or the test set-up. The temperature sensor in a DMA instrument is not in contact with the test specimen and therefore measures temperature of the environment surrounding the specimen under test. The resulting data can vary with the heating rate applied. A procedure is included to take into account the thermal lag influencing the measured data.

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 472, *Plastics — Vocabulary*

ISO 6721-1, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472, ISO 6721-1 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

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

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

3.1
temperature at peak of loss modulus curve
$T_{M''}$
temperature of the peak of the loss modulus curve vs. temperature

Note 1 to entry: It is expressed in degrees Celsius (°C).

Note 2 to entry: See Figure 1, data point 1.

3.2
temperature at inflection point of storage modulus
$T_{M'}$
temperature at inflection point of the curve of storage modulus vs. temperature

Note 1 to entry: It is expressed in degrees Celsius (°C).

Note 2 to entry: See Figure 1, data point 2.

3.3
temperature at peak of loss factor curve
$T_{\tan\delta}$
temperature of the peak in the curve of loss factor vs. temperature

Note 1 to entry: The loss factor is also called tan δ.

Note 2 to entry: It is expressed in degrees Celsius (°C).

Note 3 to entry: See Figure 1, data point 3.

3.4
zero heating rate glass transition temperature
$T_{g(0)}$
value of the glass transition temperature extrapolated to 0 K/min heating rate

Note 1 to entry: It is expressed in degrees Celsius (°C).

Note 2 to entry: See 9.2.1, Figure 2, and 9.2.2, Figure 3.

3.5
rate dependent glass transition temperature
$T_{g(n)}$
value of the glass transition temperature at n K/min heating rate

Note 1 to entry: It is expressed in degrees Celsius (°C).

Note 2 to entry: See 9.2.1 and 9.2.2.

4 Principle

A specimen of known geometry is placed or held in a suitable mechanical loading system in an enclosed temperature chamber, or oven, which can be heated at a controlled rate. The specimen is mechanically oscillated at a fixed frequency, and the resulting changes in the viscoelastic response of the material are monitored and recorded as a function of the test temperature. The dynamic properties (storage modulus, loss modulus and loss factor) are determined from the load and displacement data recorded throughout the test (see ISO 6721-1).

The glass transition temperature (T_g) is determined using one of the following characteristic points in the DMA curves:

— temperature of the peak in the curve of loss modulus vs. temperature (see 3.1);

— temperature of the inflection point of the curve of storage modulus vs. temperature (see 3.2);

— temperature of the peak in the curve of loss factor (tan delta) vs. temperature (see 3.3).

The test procedure described minimizes errors of the specimen temperature through due to the thermal lag, which varies with the heating rate used, assuming that the specimen temperature is given by the measured oven temperature.

NOTE 1 The temperature of the peak in the loss modulus curve correlates quite well with the midpoint of the storage modulus curve drawn in linear scale. For many thermoplastic materials, this temperature measured at a frequency of 1 Hz correlates well with the glass transition temperature determined as midpoint of step changes obtained in DSC curves measured at 20 K/min[3].

NOTE 2 The temperature of the inflection point in the storage modulus curve depends on the numeric representation of the storage modulus. Using a linear scale results in inflection points that are similar to glass transition temperatures obtained by DSC while plotting the storage modulus in logarithmic scale yields significantly higher values.

NOTE 3 The temperature of the peak in the curve of loss factor (tan delta) is influenced by the decrease of the storage modulus and thus usually higher than that of the peak in the loss modulus curve[3].

NOTE 4 In addition to the three methods above, the onset temperatures as defined by the intercept of the tangents below T_g and the slopes in the curves of loss modulus or loss factor (tan delta) or the decrease in the storage modulus, respectively, are also used[4].

5 Apparatus

5.1 Test equipment

The test equipment shall be capable of heating at rates from 1 K/min to 10 K/min over the required temperature range and mechanically oscillating the specimen at the reference frequency of 1 Hz or 10 Hz, respectively. The heating rate accuracy shall be ±5 % or better.

The instrument shall continuously monitor and record the sinusoidal load applied to the specimen, the corresponding sinusoidal displacement, and the phase angle as a function of the measured temperature, in order to determine the storage modulus, loss modulus and loss factor. The load and displacement capabilities of the equipment shall be sufficient for the specimens tested.

The temperature sensor should be positioned in the instrument as closely as possible to the sample under test, but ensuring it is not touching it.

The equipment shall be calibrated regularly, or when the testing mode or atmosphere is changed or when the temperature sensor is moved or changed.

5.2 Devices for measuring test specimen dimensions

These shall be in accordance with ISO 6721-1.

6 Test specimen

6.1 General

The test specimen shall be in accordance with ISO 6721-1.

6.2 Shape and dimensions

The dimensions of the specimen shall be as required by the equipment for the selected test mode.

6.3 Preparation

The preparation of the test specimen shall be in accordance with ISO 6721-1.

7 Number of specimens

This shall be in accordance with ISO 6721-1.

Prepare additional specimens (at least three) to assess the heating rate dependency of the method according to 9.2.

8 Conditioning

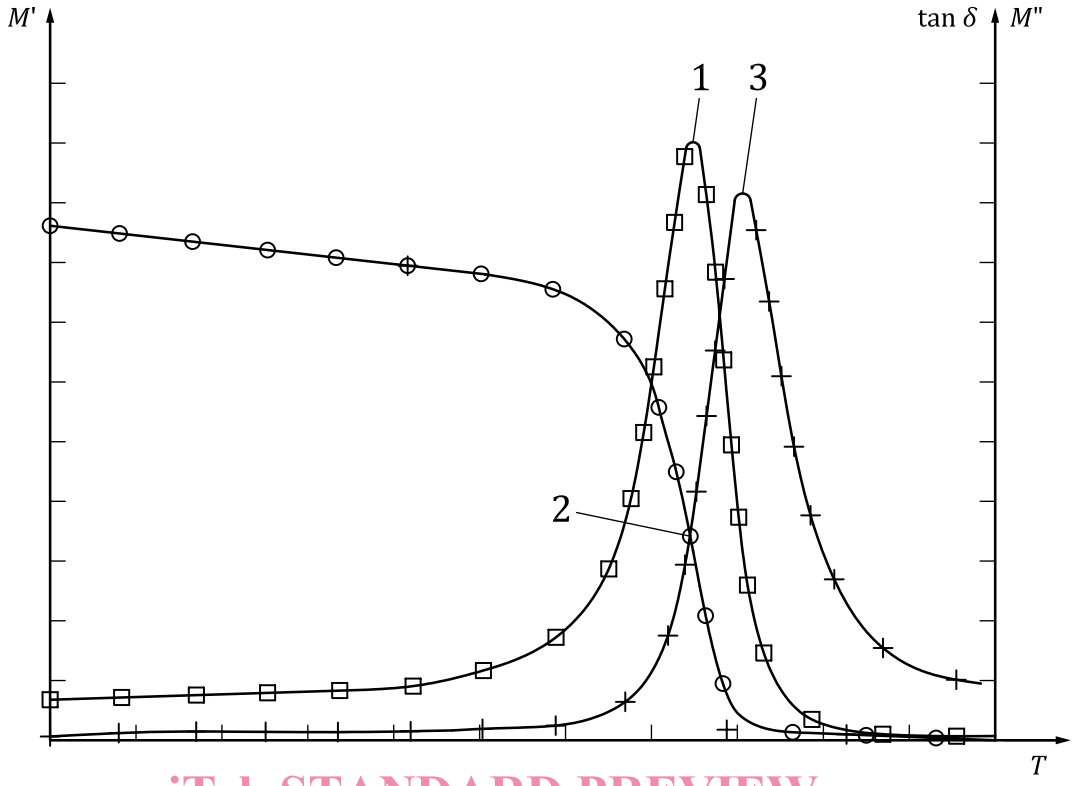
This shall be in accordance with ISO 6721-1.

9 Test procedure

9.1 Test atmosphere

This shall be in accordance with ISO 6721-1.

Measurements can be undertaken under static air conditions or an inert atmosphere. However, it is important that the calibration and the specimen tests be performed under identical conditions.



Key

1	peak of loss modulus curve, $T_{M''}$	T	temperature, °C
2	inflection point of storage modulus curve, $T_{M'}$	M'	storage modulus
3	peak of loss factor (tan δ) curve, $T_{\tan\delta}$	M''	loss modulus
—□—	loss modulus curve	tan δ	loss factor
—○—	storage modulus curve		
—+—	loss factor (tan δ) curve		

Figure 1 — Plot of dynamic mechanical data against temperature

9.2 Operation

9.2.1 Method A — Rate-dependent results — Full procedure

Mount the specimen into the instrument.

Apply a constant rate temperature scan from at least 50 K below to 50 K above the glass transition region(s) of interest at a minimum of 3 heating rates regularly spread between 1 K/min and 10 K/min. Use a new specimen for each heating rate.

For curing systems, low heating rates should be used with caution, as the data can be influenced by residual curing effects during the test resulting in overestimation of the value.

A reference test frequency of 1 Hz or 10 Hz shall be used.

The load/displacement on the specimen shall be selected so that the specimen deformation is kept within ±1 % with an accuracy of ±10 %.

Record the load and displacement data as a function of temperature, so that the storage modulus, loss modulus and loss factor can be plotted against temperature (see Figure 1). Determine the glass transition temperature using one of the methods given in Clause 4 at each heating rate.