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Rubber, vulcanized or thermoplastic — Determination of dynamic properties —

Part 3: Glass transition temperature (T_g)

Caoutchouc vulcanisé ou thermoplastique — Détermination des propriétés dynamiques —

Partie 3: Température de transition vitreuse (T_g)

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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

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Contents

Page

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 of 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 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*.

A list of all parts in the ISO 4664 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 www.iso.org/members.html.

Introduction

Elastomers are viscoelastic in nature hence their response to dynamic stress is a combination of elastic and viscous response. Glass transition temperature, T_g, is the temperature at which an amorphous or semi crystalline polymer transforms from a rubbery viscous state to a brittle glass-like state. It is always lower than the melting temperature.

This document is based on a force induced vibration test from which the stiffness can be determined, (see Annex A) and modulus and tan δ can be calculated. Tan δ is the ratio of viscous modulus to the elastic modulus. Tan δ is plotted against temperature and the glass transition temperature is taken as the peak in the curve.

The measured value of T_g depends on the experimental conditions and the mode of deformation. Measurement of T_g in dynamic mode is more sensitive to the temperature dependent physical properties of the material and is relevant to understanding its service temperature.

Rubber, vulcanized or thermoplastic — Determination of dynamic properties —

Part 3: Glass transition temperature (T_g)

1 Scope

This document specifies a method for determining the glass transition temperature, T_g, of vulcanized rubbers in the hardness range from 30 IRHD to 80 IRHD. The dynamic properties are measured via temperature sweep in sinusoidal deformation at a defined strain and frequency and T_g is determined from the peak in the tan δ versus temperature curve. Glass transition temperature, T_g, determined in this way serves the purpose of a guideline to the service temperature of the material.

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 4664-1:2011, *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 1: General guidance*

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 terms and definitions given in ISO 4664-1 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/

4 Principle

Test pieces are subjected to a temperature scan in a closed chamber at a constant strain and frequency using a dynamic mechanical analyser and the temperature of maximum tan δ taken as the glass transition.

5 Apparatus

5.1 General

The test machine used shall comply with the requirements of ISO 4664-1. Care shall be taken to avoid resonance under operating conditions.

5.2 Force capability and measurement

The force measuring device shall have an adequate range for the materials to be tested and the test pieces and deformations used. It shall enable the peak force to be read to an accuracy of ± 1,0 %.

5.3 Cycling capability

The test machine shall be able to operate at a defined frequency and with the required dynamic displacement amplitude. The deformation cycles shall be in the form of a continuous wave train of sinusoidal shape with less than 10 % harmonic content.

NOTE "Harmonic content" indicates that there is no deviation/deformation of the sine curve due to equipment contribution, sample effect or any other factors.

5.4 Damping measuring device

The test machine shall be fitted with a device to enable the loss factor of the material under test to be determined to an accuracy of ± 5 % or ± 0,02 in tan δ whichever is the smaller.

5.5 Test chamber

The test chamber shall be closed and thermostatically controlled to within ± 0,3 °C of the programmed test temperature. Care shall be taken to reduce heat conduction from the test piece through the test piece holders to the outside environment of the chamber. A means of measuring the actual temperature around the sample should be provided. Accurate temperature control is of great importance, owing to the temperature dependent properties of rubber.

5.6 Grips

Grips shall be used for clamping the test pieces in the equipment such that no slippage occurs during testing.

5.7 Measurement of test piece dimensions

Instruments to measure test piece dimensions shall be in accordance with ISO 23529.

6 Test piece

6.1 Preparation

The standard method for test piece preparation shall be direct moulding for cylinders and cut from the moulded sheet for strips or elements dedicated to the preparation of shear test pieces. If required, test pieces may be prepared from a finished article by cutting and buffing as described in ISO 23529. If the test piece is prepared from finished product, it should be free of surface irregularities, fabric layers, etc.

6.2 Dimensions

The test piece for tests in compression shall be a cylinder of 10 mm height and 10 mm diameter. Test pieces with different heights and diameters are acceptable as long as the slenderness ratio of the test piece is greater than 1.

The preferred test piece for tests in tension shall be a strip of 25 mm length, 10 mm width and 2,0 mm ± 0,2 mm thickness. The test length shall be 10 mm ± 1,0 mm with the remainder of the strip in the grips. Test pieces with different lengths, widths, thicknesses and test lengths are acceptable as long as they are in accordance with ISO 4664-1:2011, Table 2.

The test piece for tests in shear shall be of sandwich construction containing two parallel rubber elements, the thickness of the test piece shall be at least 4 times less than the diameter. The preferred thickness of the elements is 2,00 mm. In no case shall the thickness of the elements be less than 1,8 mm or more than 2,3 mm. The rubber elements shall be firmly attached to metal cylinders having diameter of 10 mm and height of 10 mm which will be held in the grip. Figure 1 provides an example of sample preparation tool and the grip.

Bonding to metal components is best done during the curing operation starting from uncured compound. When test pieces are cut from finished product, bonding with a suitable cold–setting adhesive (e.g. Loctite 407™[1)] thermal adhesive) may be used.

For shear mode analysis, all the elements in the prepared, bonded test pieces shall be aligned to the centre of axis of the metal cylinders.

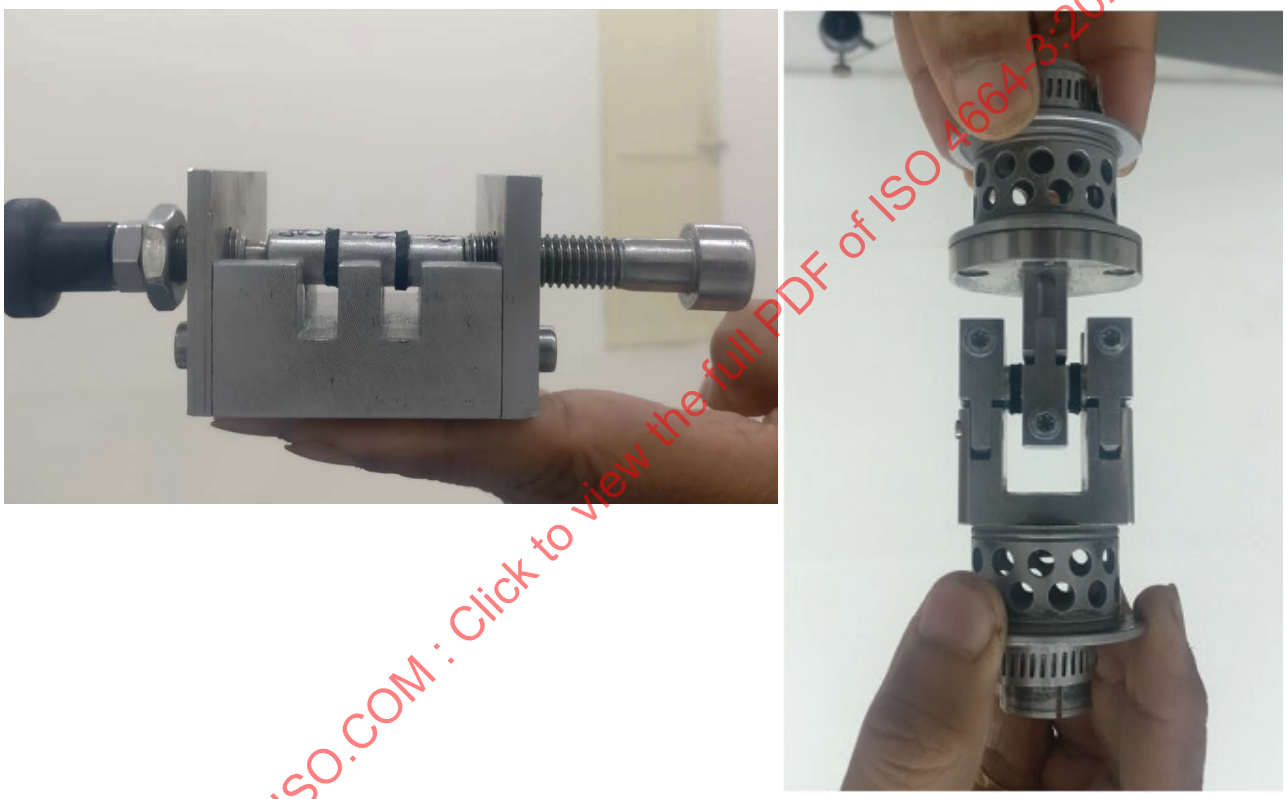


Figure 1 — Example of shear sample preparation tool and corresponding grip

7 Number of test pieces

A minimum of three test pieces shall be used.

8 Conditioning

The time interval between vulcanization and testing shall be in accordance with ISO 23529. Samples and test pieces shall be protected from light as completely as possible during the interval between vulcanization and testing.

Test pieces shall be conditioned for not less than 3 h before testing at one of the standard laboratory temperatures specified in ISO 23529.

1) Loctite 407™ is an example of a suitable product. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

9 Procedure

9.1 Measurement of the test piece dimensions

Measure the thickness of the test piece to the nearest 0,01 mm and all cross-sectional dimensions to the nearest 0,1 mm. Carry out all measurements at standard laboratory temperature.

9.2 Measurement of properties

Dynamic measurements shall be made after at least six cycles of stress have been applied to the test piece.

9.3 Test conditions

9.3.1 Strain

The strain amplitude shall be selected in accordance with the equipment manual with ± 0,01 % tolerance. Recommended values of strain amplitudes are given in ISO 4664-1 for tension, compression and shear modes.

9.3.2 Frequency

The test shall be carried out at any frequency between 1 Hz to 15 Hz. Other frequencies may be used for special purposes. Recommended frequencies are given in ISO 4664-1.

9.3.3 Temperature

If the nature of the tested compound is unknown, the test piece shall be subjected to temperature ramp starting from −150 °C to +70 °C at a rate of 1 °C/min or 2 °C/min. If the nature of the tested compound is known, start the temperature ramp at least 30 °C below its known or supposed T_g. The test chamber temperature shall be brought down to −150 °C or to the initial test temperature with the help of liquid nitrogen or any other cooling means which can maintain the temperature rise at the controlled rate of 1 °C/min or 2 °C/min.

9.4 Clamping and testing

Firmly mount or attach the test piece so that no slippage occurs during testing. Place the test piece inside the thermal chamber and lower the temperature at a rate of 8 °C/min to −150 °C (or any other defined initial temperature). Start the dynamic action.

Starting the dynamic loading before cooling is helpful to detect fracture, slipping or debonding of the test piece.

Condition the test piece at −150 °C (or any other defined initial temperature) in accordance with the time specified in ISO 23529. During the conditioning period, at least five data points should be recorded (example for conditioning period of 10 min, five data points to be recorded). After completion of conditioning period, start to raise the temperature at 1 °C/min or 2 °C/min and continuously record the data points at intervals of 10 s to 30 s.

10 Expression of results

10.1 General

All values of test piece areas and strain shall be calculated from measured values of unstrained test piece dimensions.

10.2 Calculation of tan δ

Determine the loss factor, tan δ, at all the data points recorded in accordance with ISO 4664-1 and equipment manufacturer's instructions.

Calculate the average tan δ of the three test pieces at each data point. If any single value deviates more than 10 % from the average, the test shall be repeated using two additional test pieces. Report the average of all five test pieces (average curve).

11 Determination of glass transition temperature, T_g

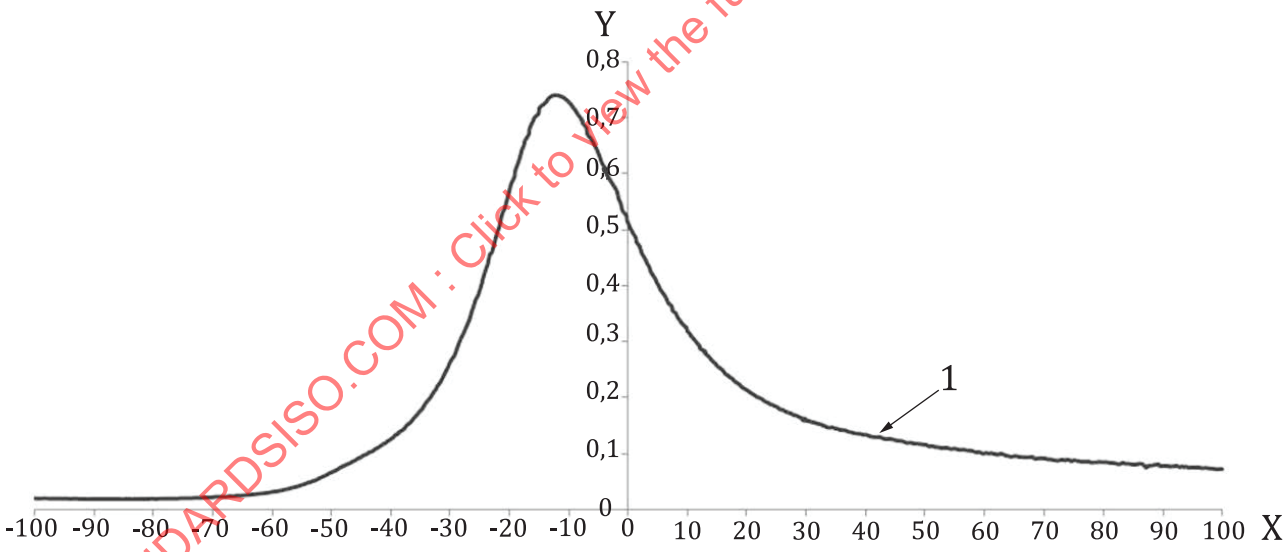
Plot the three tan δ value against the temperature. The form of the graph is as shown in Figure 2.

Option 1: Determine the tan δ value from the peak temperature of the corresponding tan δ curve; at least three values shall be determined. Report the average of three temperatures as the glass transition temperature, T_g.

Option 2: The calculation of T_g from dynamic modulus curve might not give the correct results, but if the instrument software has the option to draw the 1st order derivative of the dynamic modulus curve, then peak value of derivative can be used as T_g.

NOTE 1 In the industry, it is very common to calculate T_g from the tan δ curve.

NOTE 2 Rubber vulcanisate with more than 80 IRHD hardness will show slippage behaviour at very low temperature analysis.



Key

X temperature (°C)

Y tan δ

1 tan δ curve

Figure 2 — Example of tan δ/temperature graph

This test method operates with a limited set of conditions, includes a single frequency and single strain amplitude, to make equipment and test simple. For full assessment of the dynamic behaviour of rubber, analysis over a wide range of frequencies, strains and temperatures is required.

12 Precision

See Annex B.

13 Test report

The test report shall include following information.

a) sample details:

   1) full description of the sample and its origin;

   2) compound details and curing conditions, if known;

   3) method of preparation of test pieces from the sample, for example moulded or cut;

   4) the method of attaching test piece to the metal plates;

b) test method and test details:

   1) a reference to this document, i.e. ISO 4664-3;

   2) the test procedure used;

   3) the type of test piece used;

   4) details of the test machine including type, drive, capacity and measurement system;

   5) the test conditions, including strain amplitude, frequency and temperature scan as appropriate;

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

c) test results

   1) the number of test pieces used;

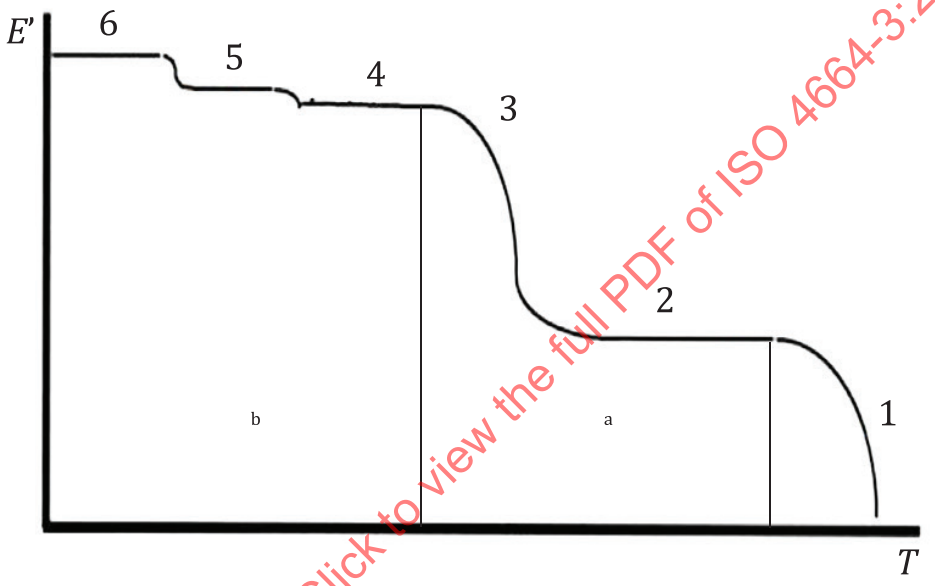
   2) the mean value of $T_g^{(\text{tested frequency})}$ (°C) of the test pieces tested; for example, T_g^{15} for T_g measured at 15 Hz.

d) the date of test.

Annex A
(informative)

Temperature dependence of dynamic properties of vulcanized rubber

The response of the vulcanized rubber when subjected to sinusoidal deformation with increasing temperature can be represented as shown in Figure A.1.



Region	6	5	4	3	2	1
Molecular motion	Localized motion	Bend and stretch bonds	Side groups	Main chain gradual mobility	Main chain large scale mobility	Chain slipping
E'	δ	γ	β	T_g (glass transition)	rubbery plateau	T_d (degradation)

Key

T temperature

E' storage modulus

a Transition zone.

b Vitreous zone.

Figure A.1 — Response of the vulcanized rubber with increasing temperature

Vitreous zone: In this zone, the macro–molecular chain movements are very limited, which results in a rigid structure and, consequently, a high modulus and a low tan δ.

Transition zone: This transition represents the beginning of the chain segment movements and is characterized by a drop in the modulus and an increase in the tan δ. The temperature at which damping is at its maximum and the modulus is the mean value between the vitreous modulus and the modulus of elasticity is called the glass transition temperature, T_g. This phenomenon is represented by the tan δ peak in the temperature scan of the cured rubber sample.

During the temperature scan, localized bond movements occur as the material warms and expands in volume. This is termed as gamma (γ) transitions. As the temperature raises, increase in free volume leads to increase in mobility of whole side chains and localized groups of atoms. This is termed beta (β) transitions. As heating continues, the amorphous region starts melting and the glass transition temperature, T_g, appears. At the end, chains start slipping and flow of material occurs. This is the degradation temperature (T_d).