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BSI Standards Publication

Aerospace series — Fibre reinforced plastics — Test method — Determination of the glass transition temperatures

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Aerospace series - Fibre reinforced plastics - Test method - Determination of the glass transition temperatures

Série aérospatiale - Matières plastiques renforcées de fibres - Méthode d'essai - Détermination de la température de transition vitreuse

Luft- und Raumfahrt - Faserverstärkte Kunststoffe - Prüfverfahren - Bestimmung der Glasübergangstemperatur

This European Standard was approved by CEN on 10 August 2013.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (EN 6032:2015) has been prepared by the Aerospace and Defence Industries Association of Europe - Standardization (ASD-STAN).

After enquiries and votes carried out in accordance with the rules of this Association, this Standard has received the approval of the National Associations and the Official Services of the member countries of ASD, prior to its presentation to CEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2016, and conflicting national standards shall be withdrawn at the latest by May 2016.

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1 Scope

This standard specifies a method to determine the apparent glass transition temperatures of non-metallic materials.

This standard is applicable to unidirectional tape and woven fabric reinforced plastic or plastic materials like adhesive or neat resin for comparison of the influence on the glass transition temperature resulting from processing-parameters of non-metallic parts, for compatibility tests for checking co-curing effects of different prepreg types or with adhesive.

This standard does not give any directions necessary to meet health and safety requirements. It is the responsibility of the user of this standard to consult and establish appropriate health and safety precautions.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies..

EN 2374, *Aerospace series — Glass fibre reinforced mouldings and sandwich composites — Production of test panels*

EN 2565, *Aerospace series — Preparation of carbon fibre reinforced resin panels for test purposes* 1)

EN 2743, *Aerospace series — Fibre reinforced plastics — Standard procedures for conditioning prior to testing unaged materials*

EN 2823, *Aerospace series — Fibre reinforced plastics — Test method for the determination of the effect of exposure to humid atmosphere on physical and mechanical characteristics* 1)

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
glass transition temperature (*T*g)
the glass transition temperature is defined for this standard as the temperature where the sample exhibits a dramatic change in mechanical and damping behaviour with increasing temperature when subjected to an oscillating displacement

Note 1 to entry: The *T*g values are determined by measuring sample stiffness (storage modulus and damping (loss modulus/tan δ) with increasing temperature using a recommended Dynamic Mechanical Analysis (DMA) instrument and evaluating the plots against temperature (see Figure 1).

3.1.1
***T*g-onset**
the *T*g-onset is defined as the temperature intersection of extrapolated tangents drawn from points on the storage modulus curve before and after the onset of the glass transition event

1) Published as ASD-STAN Prestandard at the date of publication of this standard. http://www.asd-stan.org/

3.1.2
***T*g-loss**
the *T*g-Ioss is defined as the temperature where the diagram loss modulus versus temperature has its maximum

3.1.3
***T*g-peak**
the *T*g-peak is defined as the temperature where the diagram tan δ (damping) versus temperature has its maximum

3.2
slope angle β
β is the angle of the slope of the storage modulus represented by tangent A (see Figure 1)

4 Principle of the method

Using specially designed equipment of the DMA type the storage modulus, loss modulus and the tan delta (damping) of a flat plastic sample is automatically measured and plotted while the temperature of the specimen is raised, at a defined heating rate. Plots of moduli against temperature are evaluated according to the prescribed procedures to determine *T*g-onset, *T*g-Ioss and *T*g-peak which give an indication of the maximum service temperature of the material tested.

The slope angle of the storage modulus estimates the drop of mechanical properties due to temperature effects.

4.1 Method A

The equipment is set to fixed frequency mode at 1 Hz.

4.2 Method B

The equipment is set to resonant frequency mode, initial frequency shall be 20 Hz - 50 Hz.

4.3 Method C

Freely damped torsion pendulum.

5 Designation of the method

The designation of the method used shall be drawn up according to the following example:

Description block	**Identity block**
Non-metallic materials **Glass transition temperature (*T*g)**	**EN6032 A**

Number of this standard

Method (see 4.1 and 4.3)

NOTE If necessary, the code I9005 may be placed between the description block and the identity block.

It should be stated that moduli measured by this method shall not be used for design purposes.

The results obtained by this method vary due to the equipment used. For comparing results the involved parties shall agree on the equipment used, correlation factors to compare results of different equipment and the specific material requirements.

A list of recommended equipment is given in Annex A of this standard.

When this method is involved in a material specification, with material requirements the relevant test equipment according to Annex A shall be stated.

6 Apparatus

6.1 Equipment of the dynamic mechanical analysis type with bending and/or torsional/shear loading of the specimen, capable of testing and recording frequency, storage modulus, loss modulus, tan delta and temperature in the ranges used.

Recommended instruments are given in Annex A of this standard.

6.2 Micrometer with 6 mm fiat faces and accurate to the nearest 0,01 mm.

6.3 Vernier caliper accurate to the nearest 0,1 mm.

7 Test specimen

7.1 Test specimen description

The test specimen can consist of either unidirectional tape, woven fabric or adhesive/neat resin. The fibre orientation is 0° for tape and warp for fabric.

Dimensions : Width and length of the specimen shall be chosen in accordance with the requirements of the test equipment used.

Thickness : $(2 \pm 0{,}2)$ mm (if not otherwise specified) or the nearest ply if fabric material is used.

7.2 Test specimen preparation

The specimens are cut out of plates. The coefficient of variation in thickness measurements shall be < 2 % per plate.

Where relevant the reinforced plates shall be manufactured in accordance with EN 2565 (CFRP) or EN 2374 (GFRP).

The process parameters shall be in accordance with the applicable technical specification.

Precautions shall be taken to avoid notches, undercuts, rough or eneven surfaces after machining.

7.3 Number of test specimens

Three specimens shall be tested per test condition, except when otherwise specified in the applicable technical specification. If tests are carried out after ageing or at a temperature different from room temperature, care should be taken to ensure that room temperature/dry reference specimens which have been machined from the same plate as the specimen under investigation are also tested.

7.4 Ageing of specimen

ln case of tests after exposure to humid atmosphere, the conditioning shall be according to EN 2823.

8 Procedure

8.1 Conditioning

Prior to test cured specimens shall be stored at (23 ± 2) °C and (50 ± 5) % relative humidity in accordance with EN 2743. Aged specimens shall be tested directly after the ageing procedure (a maximum delay of 8 h at (23 ± 2) °C is allowed).

8.2 Determination of dimensions

Before ageing and mechanical testing measure and record the thickness and width at 3 points in the non-gripping area of the specimen. Use for the thickness the micrometer (see 6.2) and for the width the vernier caliper (see 6.3) or the micrometer (see 6.2).

8.3 Calibration

The equipment shall be calibrated according to the manufacturers instructions.

The temperature calibration shall be carried out using *T*g-Ioss peaks of agreed grades of:

— Polycarbonate : *T*g-Ioss, (153 ± 1) °C.

— Polyethersulphone : *T*g-Ioss, (221 ± 1) °C.

8.4 Testing

The relevant test parameters shall be used:

— Temperature : Room temperature to, at least, 20 °C higher than point L or M, see Figure 1.

— Heating rate : (5 ± 0,2) °C/min (3 °C/min optional).

— Specimen dimensions : Actual.

Clamp the specimen firmly in the sample holder of the equipment used according to the manufacturers instructions.

Perform the test according to the manufacturers instructions.

9 Presentation of the results

9.1 Diagram

The test results shall be plotted in a diagram according to Figure 1, showing storage modulus, loss modulus and tan delta (optional) versus temperature.

9.2 Determination of *T*g-onset

The *T*g-onset shall be determined by introducing two tangents into the diagram of the storage modulus curve, see Figure 1.

— The first one (tangent A) representing the linear area of the curve from the start temperature up to the beginning of the dramatic slope of the curve.

— The second one (tangent B) shall be the tangent left of the point of inflection (= *t*g-Ioss) on the decreasing part of the curve (see typical diagram Figure 1).

— Both tangents shall be extended to intersect in point C.

— The temperature related to point C shall be the *T*g-onset.

9.3 Determination of *T*g-loss

*T*g-Ioss is the temperature which corresponds to the maximum of the loss modulus curve (point L in Figure 1)

9.4 Determination of *T*g-peak (optional)

*T*g-peak is the temperature which corresponds to the maximum tan-δ (point M in Figure 1).

9.5 Determination of the slope angle β (optional)

Evaluate the slope of the storage modulus curve which is represented by tangent A according to 9.2 (see Figure 1). Determine β by the given formula:

$$\beta = \text{arc tan} \frac{\Delta E'}{\Delta T}$$

9.6 Quantitative check

*T*g results should be within ± 2,0 °C of their mean. If greater, the results should be scrutinized to see if they are acceptable.

10 Test report

The test report shall refer to this standard and include the following:

10.1 Complete identification of the material tested, including at least material designation, supplier, batch number, roll number, fibre areal weight, filament count, processing details, stacking sequence, test orientation.

10.2 All details regarding specimen preparation including cure parameters.

10.3 The measured specimen dimensions.

10.4 Ageing and/or exposure condition prior to the test.

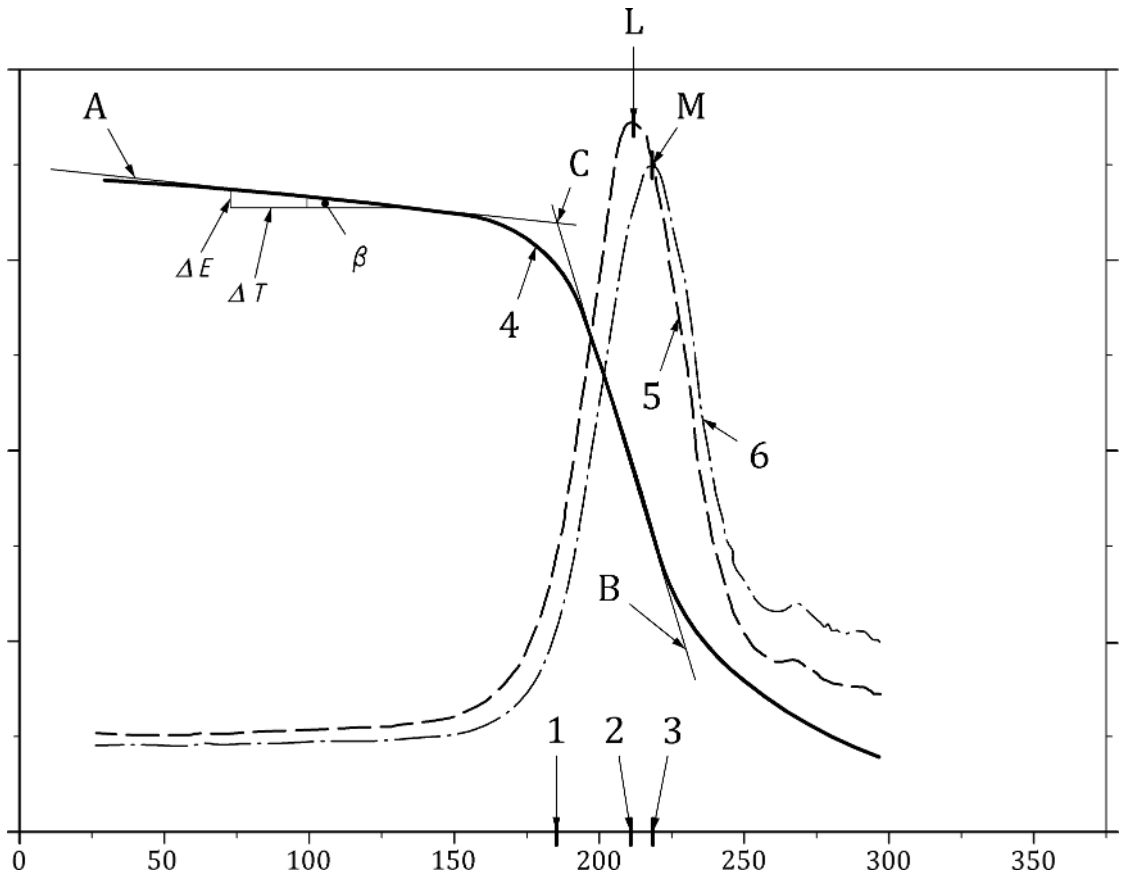
10.5 Date of test, facility and identification of individuals performing the tests.

10.6 Equipment, method and test parameters used including: Displacement amplitude, strain frequency, strain rate.

10.7 Plot of storage modulus, loss modulus and tan delta (optional) versus temperature, the tangents A and B and the points C, L and M according to Figure 1.

10.8 Individual test results of *T*g-onset, *T*g-Ioss, *T*g-peak (optional) and β (optional).

10.9 Any incident which may have affected the results and any deviation from this standard.



Key

1 *T*g-onset
2 *T*g-loss
3 *T*g-peak
4 *E′* (GPa) Storage Modulus
5 *E″* (GPa) Storage Modulus
6 Tan Delta

Figure 1

Typical diagram storage modulus, tan delta and loss modulus versus temperature determination of *T*g-onset, *T*g-peak, *T*g-loss and *β*.

Annex A
(informative)
Equipment

The following equipment may be used to determine Glass Transition Temperature.

A.1 PL-DMTA dynamic Mechanical Thermal Analyser
The Technology Centre
Epinal Way
Loughborough
LE 11 OQE
U.K.

A.2 981/982/983 DMA Dynamic Mechanical Analyser
TA Instruments
Silver Side Road
Wilmington,
U.S.A.

A.3 A.T.M. 3 torsion Pendulum,
Myrenne GmbH
5106 Roetgen
Federal Republic of Germany

A.4 Torsiomatic torsion Pendulum,
Zwick GmbH + Co
7900 Ulm
Federal Republic of Germany

A.5 RDS 7700
Rheometrics Inc.
2438 US Hwg 22
Union
NJ 07083
USA

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