
**Aeronavtika - Z vlakni ojačeni polimerni materiali - Preskusna metoda -
Ugotavljanje temperaturne prehodnosti stekla**

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

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

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

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EUROPEAN STANDARD
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Determination of the glass transition temperatures**

Série aéronautique - Matières plastiques renforcées de
fibres - Méthode d'essai - Détermination de la
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Prüfverfahren - Bestimmung der
Glasübergangstemperatur

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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of an identical text or by endorsement, at the latest by May 2016, and conflicting national standards shall be withdrawn at the latest by May 2016.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/ $\tan \delta$) 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*-loss 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 $\tan \delta$ (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*-loss 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 (<i>T_g</i>)	<u>EN6032 A</u>
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.

EN 6032:2015 (E)

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 flat faces and accurate to the nearest 0,01 mm.

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

7 Test specimen

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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 uneven 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

In 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 $(23 \pm 2) ^\circ\text{C}$ and $(50 \pm 5) \%$ relative humidity in accordance with EN 2743. Aged specimens shall be tested directly after the ageing procedure (a maximum delay of 8 h at $(23 \pm 2) ^\circ\text{C}$ 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 -loss peaks of agreed grades of:

— Polycarbonate : T_g -loss, $(153 \pm 1) ^\circ\text{C}$.

— Polyethersulphone : T_g -loss, $(221 \pm 1) ^\circ\text{C}$.

8.4 Testing

The relevant test parameters shall be used:

- Temperature : Room temperature to, at least, $20 ^\circ\text{C}$ higher than point L or M, see Figure 1.
- Heating rate : $(5 \pm 0,2) ^\circ\text{C}/\text{min}$ ($3 ^\circ\text{C}/\text{min}$ 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.