SLOVENSKI STANDARD
SIST EN 6064:2018

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Aeronavtika - Analiza nekovinskih materialov (obdelanih) za določanje obsega obdelave z diferenčno dinamično kalorimetrijo (DSC)

Aerospace series - Analysis of non-metallic materials (cured) for the determination of the extent of cure by Differential Scanning Calorimetry (DSC)

Luft- und Raumfahrt - Analyse von nichtmetallischen Werkstoffen (gehärtet) zur Bestimmung des Vernetzungsgrades durch dynamische Differenzkalorimetrie (DSC)

Série aérospatiale - Analyse Enthalpique Différentielle (AED) des matériaux non métalliques (polymérisés) pour la détermination du degré de polymérisation

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Aerospace series - Analysis of non-metallic materials (cured) for the determination of the extent of cure by Differential Scanning Calorimetry (DSC)

Série aérospatiale - Analyse Enthalpique Différentielle (AED) des matériaux non métalliques (polymérisés) pour la détermination du degré de polymérisation

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

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European foreword

This document (EN 6064:2017) 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of an identical text or by endorsement, at the latest by June 2018 and conflicting national standards shall be withdrawn at the latest by June 2018.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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1 Scope

This test method defines the procedure for the estimation of the extent of cure of certain non-metallic materials (e.g. preimpregnated and neat resin systems, adhesives) for aerospace use. The extent of cure is estimated by Differential Scanning Calorimetry (DSC) measurements of uncured (reference) and cured materials. Additional evidence on the extent of cure may be gained by combining results from this method with those obtained by other techniques.

This standard does not give any directions necessary to meet the health and safety requirements. It is the responsibility of the user of this standard to adopt 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 2331, *Aerospace series — Textile glass fibre preimpregnates — Test method for the determination of the resin and fibre content and mass of fibre per unit area*

EN 2559, *Aerospace series — Carbon fibre preimpregnates — Determination of the resin and fibre content and the mass of fibre per unit area*

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

3 Symbols and definitions

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

The determination of these parameters shall be agreed upon between manufacturer and purchaser in case of non-ideal curves or different instrument types.

3.1
curing reaction (see Figure 1)

θ is the heating rate, in degrees Celsius or Kelvin by minutes;

ΔH is the reaction enthalpy, in Joules by grams;

ΔH_{100} is the reaction enthalpy corrected to 100 % resin content, in Joules by grams;

A-curve is the reference reaction curve for uncured material;

B-curve is the reaction curve for an already (semi-) cured sample.

3.2
calibration (see 6.6)

T_m is the melting temperature, in degrees Celsius or Kelvin

ΔH_m is the enthalpy of fusion, in Joules by grams

4 Principle of the method

Differential Scanning Calorimetry (DSC) measures the temperatures and the heat flow associated with transitions in materials as a function of time and temperature.

5 Designation of the method

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

Description block	Identity block
ANALYSIS OF NON-METALLIC MATERIALS DETERMINATION OF THE EXTENT OF CURE BY DIFFERENTIAL SCANNING CALORIMETRY (DSC)	EN6064

Number of this standard

6 Apparatus

6.1 The test shall be performed in a Differential Scanning Çalorimeter (DSC) capable of operating within the limits laid down in the test procedure.

Instruments are given in the Annex A of this standard.

If the DSC-apparatus of the manufacturer and the purchaser are different, a cross-check for comparison of the test results is necessary.

6.2 Balance with an accuracy of 0,01 mg.

6.3 Ancillary items such as sharp cutting knife and tweezers.

6.4 Nitrogen (dry, min. 99,99 % purity).

6.5 Hollow punch with a diameter 1 mm < the diameter of the sample pan to cut out the required discs of adhesive, prepreg or laminate.

6.6 Calibration

The equipment shall be controlled using a pure indium sample to maintain a temperature accuracy to ± 0,5 °C and a calorimetric precision within 1 % during test. Periodically the temperature and cell constant shall be calibrated using pure samples of the materials specified in Table 1 (type and values according to DSC manufacturer recommendations).

Temperature calibration shall be performed using two or preferably three materials over the temperature range of interest.

The heating rate for calibration shall be the same as used in the subsequent experiments i.e. 10° C/min for standard operation (see Clause 8).

The instrument calibration shall be performed using the same sample pan type, purge/purge rate and equivalent thermal mass samples as will be used in subsequent experiments.

The base line and the cell shall be checked periodically (interval to be agreed between the parties). The base line drift for a pair of empty pans or pans containing an inert substance shall not exceed 1,0 mW over the range – 50 °C to 300 °C.

7 Test specimens

7.1 Preparation

7.1.1 General

Test specimens shall be selected in accordance with the sampling procedure defined in the Material Specification. For the A-curve uncured samples, for the B-curve (semi-) cured samples shall be taken.

Each sample shall comprise approximately the equivalent of 7 mg to 10 mg of neat resin (for example 20 mg of prepreg). In case of extreme enthalpy of reaction the sample size of the material may be adjusted to achieve acceptable curves in the diagram.

The sample mass shall be determined to 0,01 mg (see 6.2). For quantitative measurements it is necessary to correct the ΔH to 100 % resin content (ΔH_{100}), the resin content shall be determined using the test methods of 7.1.2 or 7.1.3.

A minimum of two test specimens shall be used for standard operations (e.g. release testing). For determination of standard deviations a minimum of six specimens are necessary (e.g. qualification programs).

7.1.2 Determination of actual fibre/resin content

Carbon composite : according to EN 2559;

Glass composite : according to EN 2331;

Aramid composite : according to EN 2559 but using HNO_3/DMSO.

If an insoluble filler (such as SiO_2) is present, it shall be determined separately (after the removal of the fibre) by centrifuging and its mass added to the reinforcement content.

7.1.3 Determination of actual carrier/resin content for adhesives (uncured)

Usual extraction technique: use ultrasonic bath and appropriate solvent such as DMF, DMSO, NMP.

Determine the carrier weight after twice flushing with solvents such as Acetone or MEK and thorough drying.

If an insoluble filler is present, it shall be determined separately (after the removal of the carrier) by centrifuging and its mass added to the reinforcement content.

Actual resin content: $m_{resin} = m_{adhesive} - m_{carrier}$.

7.2 Conditioning

7.2.1 Conditioning of material stored at ambient temperature

For material stored at ambient temperature, the amount of material required for testing shall be sampled and conditioned in the standard atmosphere (defined in EN 2743) for a minimum of 2 h, unless otherwise specified.

7.2.2 Conditioning of material stored below ambient temperature

For material stored at temperatures lower than ambient temperature, the material, suitably packed in an airtight bag (containing a suitable desiccant) to prevent moisture pick-up, shall be allowed to reach ambient temperature over a period of time according to the mass of the package.

The actual time shall be recorded in the report. When the material has reached ambient temperature, the amount (together with the release film on it) required for testing shall be sampled and conditioned in the standard atmosphere (defined in EN 2743) for a minimum of 2 h, unless otherwise specified.

7.2.3 Standard atmosphere for testing

The test equipment shall be at (23 ± 2) °C and (50 ± 5) % relative humidity (EN 2743 B conditions).

The actual test environment is controlled inside the equipment.

7.2.4 Time interval between conditioning and testing

After conditioning, the test shall be carried out within 6 h, unless otherwise specified, the specimen being kept in the standard atmosphere until the test is carried out.

8 Procedure

8.1 A-curve

The sample shall be placed in a sample pan ensuring that it is in good thermal contact with the pan. The lid shall be firmly crimped on but not hermetically sealed to avoid pressure increase. Place the two pans containing the sample and the reference into the DSC cell (see 6.1), close the system and purge with pure dry nitrogen (preferred flow 20 ml/min to 30 ml/min).

The following test conditions shall apply:

— reference sample empty sample pan and lid (if necessary containing inert material for example alumina);

— atmosphere Nitrogen (see 6.4);

— test start temperature room temperature;

— test end temperature After the end of the region of interest;

— heating rate 10 °C/min standard, unless otherwise specified;

— a record of heat flow (dq/dt) versus temperature shall be obtained between test start and test end.

8.2 B-curve

Follow 8.1, but use samples cured in an DSC pan eitherusing the standard curing cycle or the cycle to be investigated. For investigation of manufactured parts or laminates the samples shall be taken from the item concerned.