INTERNATIONAL STANDARD

ISO 22821

First edition
2021-01

Carbon-fibre-reinforced composites — Determination of fibre weight content by thermogravimetry (TG)

Composites renforcés de fibres de carbone — Détermination de la teneur en masse de fibres par thermogravimétrie (TG)

Reference number
ISO 22821:2021(E)

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

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

Methods for the determination of fibre content of carbon fibre reinforced plastics has been established in ISO 14127. The fibre mass in the composite sample is derived by removing the plastic/polymer part in the sample by combustion using burner and the use of strong acid in ISO 14127. These methods are not recommended on the grounds of safety and reagent waste. The determination method of the content of carbon black in the rubber and rubber products is regulated by ISO 9924-3. A thermogravimeter is employed as the apparatus to remove the rubber part of the sample in ISO 9924-3. Currently, thermogravimeters are produced commercially with accuracy, repeatability and reproducibility sufficient for the determination of fibre content in carbon fibre reinforced plastics. In this document, a thermogravimeter is used as the apparatus to remove the plastic/polymer part of the composite sample.

Carbon-fibre-reinforced composites — Determination of fibre weight content by thermogravimetry (TG)

1 Scope

This document specifies a thermogravimetric method for the determination of fibre weight content by weight percent, of carbon fibre reinforced composites.

This method applies to pre-products, such as, prepregs, parts and products of carbon fibre reinforced composites.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

3 Terms and definition

For the purposes of this document, the following terms and definitions 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
fibre weight content
w_f
<fibre based composites> ratio of fibre weight to total weight of composite, same as fibre content by weight; expressed as a percentage

4 Principle

A weighed test specimen is heated following a pre-set programme in a known atmosphere.

Initial pyrolysis in an inert atmosphere (nitrogen) is followed by combustion in an oxidizing atmosphere.

Generally, the reactions that generate mass variations are decompositions, oxidations, or reactions volatilizing a constituent.

Plotting the loss of mass as a function of temperature gives a quantitatively usable thermogram, which is characteristic of the material.

5 Reagents

5.1 Nitrogen gas, of minimal purity 99,995 % mass fraction, with an oxygen content of less than 10 mg/kg (ppm) and hydrocarbon content less than 1,5 mg/kg (ppm).

5.2 Dry air, with no detectable trace of oil.

The air used may be reconstituted nitrogen and oxygen of purity minimum 99,5 % mass fraction. In some cases, pure oxygen may be used.

6 Apparatus — Thermogravimetric analyser

6.1 General

Various types of analyser are commercially available. The basic components of an analyser are listed in 6.2 to 6.8.

6.2 Thermogravimetric balance, comprising a microbalance provided with a pan made from a non-oxidizable material, that can weigh up to 50 mg, is readable to the nearest 1 µg, and equipped with an oven capable of being maintained at temperatures from room temperature to approximately 1 000 °C.

6.3 Furnace, allowing the sample to be heated under a specified atmosphere and temperature.

6.4 Pan or crucible, with a size suitable to accommodate the sample and small enough to reduce the influence of buoyancy, and without melting to 1 000 °C.

Pan or crucible made of platinum or almina is recommended .

6.5 Temperature-control system, allowing heating rates to be controlled from 10 °C/min to 50 °C/min.

6.6 Gas selector, allowing successive introduction of the inert gas and oxidizing gas while controlling the flow rate.

6.7 Flow valve control and meter, for controlling gas flow rate in the range 10 cm^3/min to 250 cm^3/min.

6.8 Data acquisition and processing system, allowing temperature and weight data to be recorded and to be analysed during all the operating steps.

7 Preparation of samples

7.1 Conditioning of samples

Test samples should be conditioned in standardized laboratory conditions of temperature and humidity. A quantity of material sufficient to complete the tests is taken as the test sample and conditioned for a sufficient time to re-establish temperature equilibrium. This conditioning shall be carried out in one of the standard atmospheres specified in ISO 291.

7.2 Test specimen

Prepare a test specimen of 10 mg ± 2 mg mass cut it as a single piece. They shall be less than 1 mm in thickness and shall be 3 mm to 5 mm in length and width. A number of test specimens should be prepared according to the type of carbon fibre-reinforced plastic type of and purpose of measurement.

The test portion should be carefully prepared, since the test portion can influence the kinetics of the phenomena.

A number of test pieces depend on the type of CFRP or purposes of measurement. Standard deviation of fibre content measured by thermogravimeter for type of CFRP with weave clothes is almost the same as that measured by combustion (see Annex B). Three samples or more can be enough for CFRP with weave clothes. More than five of test portions can be required for the analysis of distribution of fibre content or CFRP with short fibres.

8 Test procedure

8.1 Description of the operating steps

Table 1 gives details of the operating steps for the procedure.

Table 1 — Operating steps

	Operating steps	Control limits	Units
1	Initial temperature	35 ± 10	°C
2	Heating rate under nitrogen	10 or 20	°C/min
3	Target temperature under nitrogen	650	°C
4	Dwell time at target temperature under nitrogen	5 or 10	min
5	Cooling under nitrogen	650 to 400	°C
6	Temperature at the change of atmosphere	400	°C
7	Dwell time at atmosphere change temperature under air	10 or 15	min
8	Heating rate under air [a]	10 or 20	°C/min
9	Final temperature under air according to the equipment [b]	800 to 850	°C
10	Maintenance time at the final temperature under air	10 to 20	min

[a] Fix the value for the heating rate under nitrogen and air: choose 10 or 20 and keep it constant during the duration of the test.

[b] If procedures do not result in a thermogram that achieves constant mass at final temperature under air, maintain the final temperature condition until constant mass is achieved.

8.2 Test processes

8.2.1 Connect the apparatus and adjust (6.6) the gas flow to a rate between 20 cm^3/min and 250 cm^3/min (6.7). Set the parameters according to the chosen process.

In the absence of equipment manufacturer' recommendation, use a flow rate of 100 cm^3/min.

8.2.2 Before the test, ensure that the pan (6.4) or the crucible is clean and empty.

8.2.3 Close the thermogravimetric balance oven (6.2), purge with a nitrogen (5.1) flow at the preset rate. Wait until stabilization. Adjust the zero to compensate for the mass of the pan or the crucible.

8.2.4 Place the test piece prepared in accordance with Clause 7 in the pan or the crucible and weigh it under the conditions specified in 8.2.3. Record the mass, m_0.

8.2.5 Conduct the test by following the operating steps specified in Table 1.

8.2.6 At the end of the test, allow the oven to cool to room temperature, open it and clean the pan or the crucible.

9 Expression of results

9.1 Data recordings and analyses

Make two different types of recording to enable the necessary calculations to be made:

a) a plot of the variation in mass, (where m_0, is the initial mass) m, vs. temperature (T) or time (t);

b) a derivative plot, dm/dT or dm/dt.

These will be used to derive the content of the various matrix resin.

9.2 Determination of fibre content

Example of a thermogram is given in Annex A.

The derivative plot shall be used to define particular points on the mass variation plot as follows. Identify on the derivative plot the minima A'_0, A'_1, A'_2, and, A'_3 corresponding to the closest points to return to zero of the derived function. Note these points on the main curve of mass change. Report A_0, A_1, A_2, and A_3 on the ordinate and read the corresponding masses m_0, m_1, m_2, and m_3.

The percentage mass fraction loss, w_f, due to carbon fibre combustion is given by Formula (1):

$$w_f = \frac{m_1 \times m_2}{m0} \times 100 \quad (1)$$

where

m_0 is the initial mass of the test piece;

m_1 is the mass of the test piece remaining after pyrolysis;

m_2 is the mass of the test piece remaining after pyrolysis and carbon fibre combustion;

This method should be applied to the composites, when it is confirmed that the most of the resin of the composites can be thermally degraded reaching 650 °C under nitrogen gas by thermogravimetry analysis.

NOTE 1 These operations can be calculated by computer.

NOTE 2 The fibre volume content and the void content can be calculated by using the formulae regulated in ISO 14127[3].

10 Precision

See Annex B.

11 Test report

The test report shall contain at least the following information:

a) sample details:

1) full description of the sample and its origin,

   2) conditions of preparation of the sample;

b) test method:

   1) a full reference to the test method used, i.e. the number of this document (ISO 22821:2021);

c) test details:

   1) the laboratory temperature,

   2) the time and temperature of conditioning prior to test,

   3) the temperature of test, if other than standard laboratory temperature,

   4) type of equipment used,

   5) the heating rate

   6) the flow rate

   7) details of any procedures not specified in this document as well as the possible incidents;

d) test results:

   1) the number of test pieces used,

   2) the individual test results,

   3) the date of test.

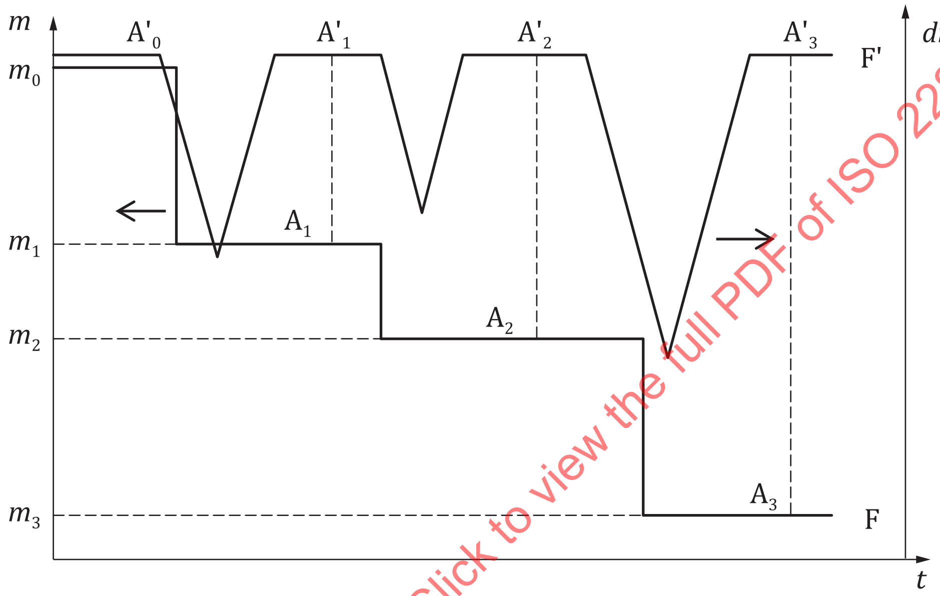
   4) operator name

Annex A
(informative)

Example of thermogram

Examples of various thermograms are illustrated in Figures A.1 to A.3.

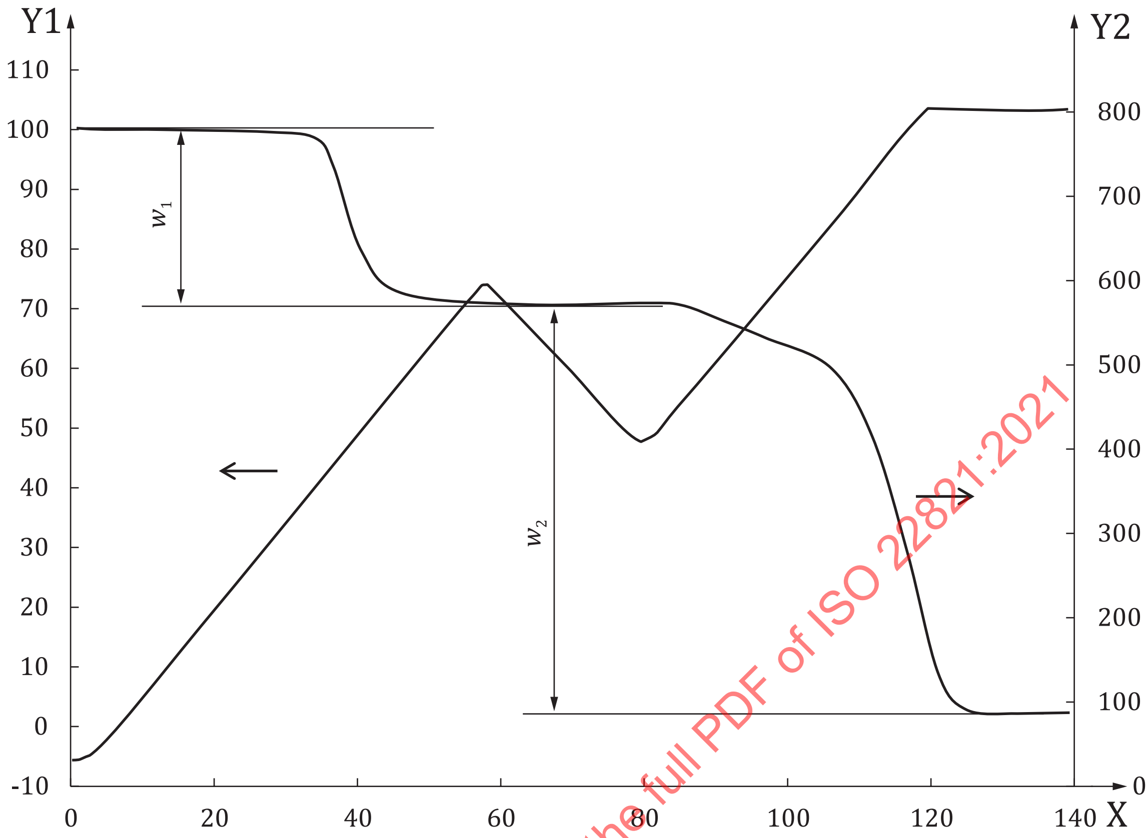
An example of a thermogram of carbon fibre reinforced epoxy resin, consisting of carbon fibre reinforced thermoset plastics (CFRP), using thermogravimetry is shown in Figure A.1.



Key

A0, A1, A2, A3	absolute minima
A'0, A'1, A'2, A'3	derivative minima
F	main curve
F'	derivative curve
m	mass, in mg
m_0	initial test piece mass
m_1	test piece mass after pyrolysis
m_2	test piece mass after pyrolysis and carbon fibre combustion
m_3	residue yield mass
t	time, in min
dm/dt	derivative plot, in mg/min

Figure A.1 — Terminal diagram



Key

X time min

Y_1 weight fraction %

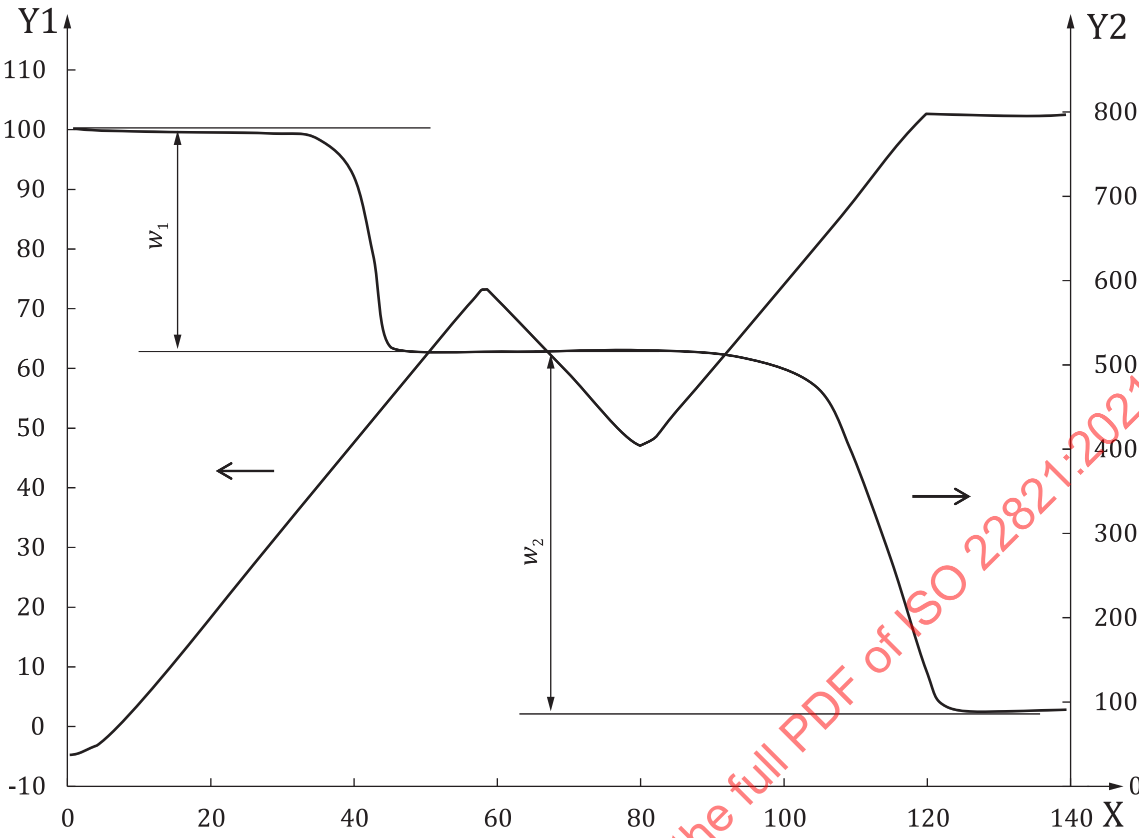
Y_2 temperature °C

w_1 content lost due to pyrolysis of plastics

w_2 content lost due to combustion of carbon fibre

Figure A.2 — Example of thermogram for carbon fibre reinforced thermoset (CFRP)

An example of a thermogram of carbon fibre reinforced epoxy resin, using thermogravimetry is shown in Figure A.2. It is observed that the weight fraction gradually decreases in a range time 90 min and 100 min. Some part of carbon-fibre may degrade under air.



Key

X time min

Y_1 weight fraction %

Y_2 temperature °C

w_1 content lost due to pyrolysis of plastics

w_2 content lost due to combustion of carbon fibre

Figure A.3 — Example of thermogram for carbon fibre reinforced thermoplastics (CFRTP)An example of a thermogram of carbon fibre reinforced polyamide, using thermogravimetry is shown in Figure A.3.