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Carbon fibres — Determination of polyacrylonitrile-based (PAN-based) carbon fibre tow characteristics — Heat transfer parameter

Fibres de carbone — Détermination des caractéristiques du câble en fibres de carbone à base de polyacrylonitrile (PAN) — Paramètre de transfert de chaleur

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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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Foreword

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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

Currently, contact-type (1) and noncontact-type thermal conductivity method (2) are used to measure the thermal conductivity. The contact-type method exhibits measurement non-uniformity in the carbon fibre thickness direction to unable uniform sampling, and the error occurrence rate is known to be high. Also, the noncontact-type method, fibre porosity and tow gap can create lower accuracy, and the error occurrence rate is similarly high. Therefore, this document is made to provide a quantitative method for the measurement of carbon fibre thermal conductivity, using a thermal imaging camera, that will alleviate problems observed in prior described methods.

Carbon fibres — Determination of polyacrylonitrile-based (PAN-based) carbon fibre tow characteristics — Heat transfer parameter

1 Scope

This document specifies a method using a thermal imaging camera for measuring the heat transfer parameter of PAN-based 12 K carbon fibre tow with a filament diameter of 7 µm. This document is applicable to both sized and unsized carbon fibres.

NOTE At the time of publication, the experience is on 12 K tow. Other tows will be included when the experience becomes available.

2 Normative references

There are normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions are 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
carbon fibre
fibrous carbon materials having a mass content of carbon element of 90 % or more, obtained by pyrolysis of organic materials

3.2
carbon fibre tow
untwisted bundle of continuous carbon fibre filaments

Note 1 to entry: The tow is provided in a wide variety of sizes from 1 K, 3 K, 6 K, 12 K, 24 K, etc. 12 K indicates 12 000 filaments. The 12 K carbon fibre tow used in this experiment has filaments with about 7 µm in diameter.

3.3
polyacrylonitrile
PAN
polymer of acrylonitrile

Note 1 to entry: PAN is a raw material used for the carbon fibre production.

[SOURCE: ISO 472:2013, 2.721, modified — A note to entry has been added.]

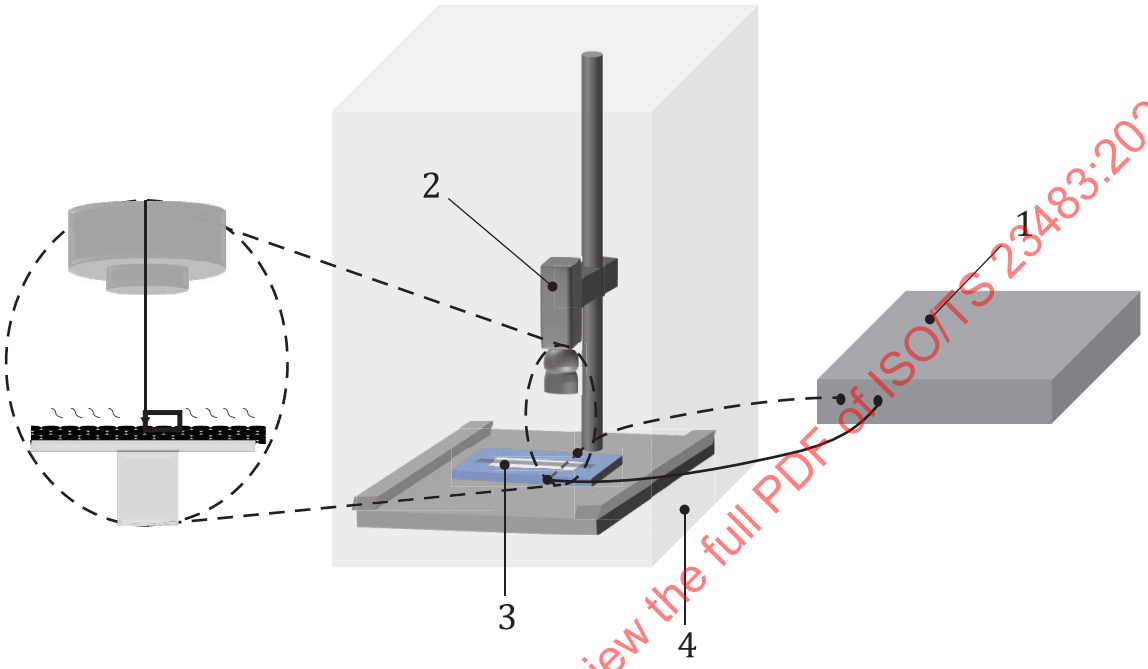
4 Principle

A thermal imaging camera is used to measure the temperature difference of the carbon fibre tow from one side of electrode A to the other side of electrode B (distance of 15^{+5}_{-0} mm) between which the heating is measured (see Figure 2.). According to the heating power and the cross-sectional area of the tow, the

heat transfer parameter of the carbon fibre tow is calculated by the Fourier's thermal conductivity law in 8.7.

5 Test devices

The test equipment shown in Figure 1 consists of thermal image camera, camera tripod, sample holder, and power supply.



Key

1 DC power supply
2 infrared thermal imaging camera
3 carbon fibre tow specimen
4 adiabatic black box chamber

Figure 1 — A typical diagram of carbon fibre tow heat transfer parameter measurement system

5.1 Thermal image camera, camera which can measure temperature gradient appeared on sample surface.

5.1.1 Resolution

The minimum requirement on resolution of the thermal image camera shall be 640 × 480 and the accuracy is within ±275 K of reading.

5.1.2 Emissivity

Emissivity is the total amount of thermal energy emitted per unit area per unit time for all possible wavelengths and is a value between 0 and 1. For carbon (graphite), emissivity value at 298 K is 0,97[3].

NOTE One of the most important conditions when measuring the temperature using a thermal image camera is the emissivity of the object to be measured. The emissivity determines how accurately the temperature can be measured. Therefore, each material has its unique emissivity value.

5.2 Sample holder, which consists of two copper electrodes, insulation cover, and clamps which are used to fix the carbon fibre tow in a straight line (see Figure A.1.) One of the electrode (see A in

Figure 2.) from which heat is transferred to the carbon fibre tow shall be a fixed part and the other electrode, B, shall be made of a moving part so as to enable adjustment the length of the carbon fibre tow located between the electrodes. When the carbon fibre tow is mounted in a straight line between two electrodes, tow is fixed with clamps at positions with electrode having insulation cover.

5.3 Power supply, a DC power supply having 50 V, 20 A with constant supply of 1 000 W power shall be used.

6 Test conditions on carbon fibre tow

When measuring heat transfer parameter, the temperature and the relative humidity shall be maintained at (296 ± ^{2}) C and (50 ± 10) %, respectively. The surroundings shall be maintained so as to not affect the temperatures being measured.

Convective chambers shall not be as the convective air can affect the carbon fibre tow temperature. A simple black box chamber has been shown to be affective in isolating the carbon fibre tow temperature from the surroundings.

7 Traceability — Calibration

The thermal imaging camera shall be calibrated once a year in accordance to the manufacture's recommendations [standard calibration procedure for radiation thermometers (CP801-50204-1) and the National Metrology Institute].

8 Test procedures

8.1 Type

PAN-based 12K carbon fibre tow shall be used for measuring heat transfer parameter.

8.2 Size

The length of the carbon fibre tow should be 100^{+5}_{-0} mm.

NOTE The total length of the carbon fibre tow does not have any significance and 100 mm length is chosen for the purpose of handling during the test.

8.3 Thermal imaging camera

8.3.1 Working parameters of thermal imaging camera

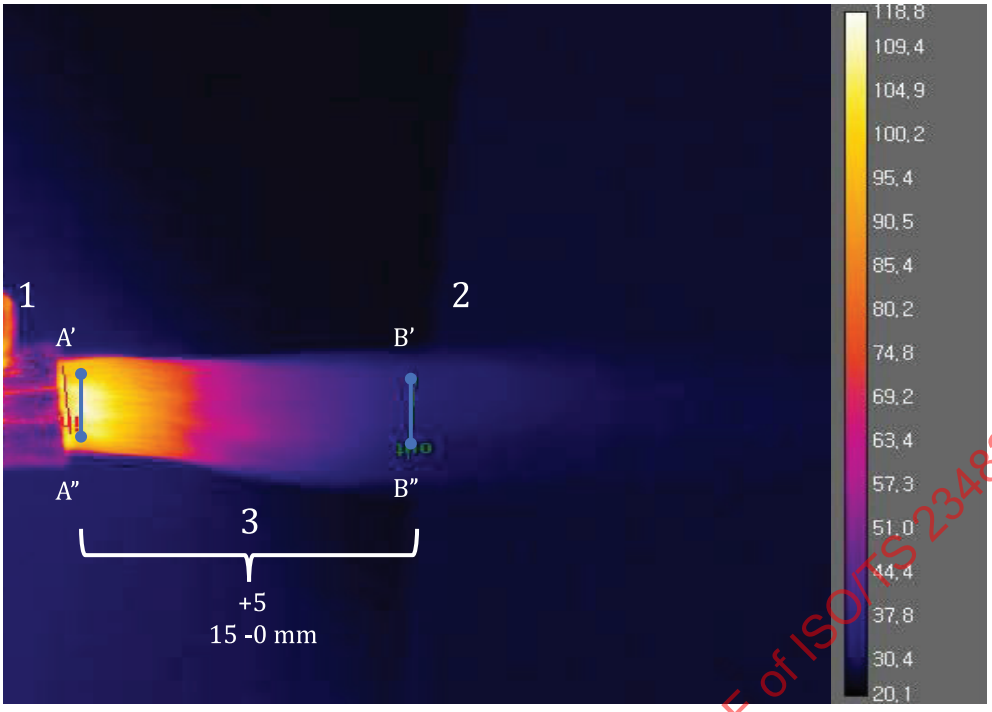
When measuring the thermography image of the fibre with a thermal imaging camera, the temperature value of the sample is providing (emissivity, distance to the sample, and sample area) as camera correction values.

8.4 Thermal imaging camera scan range on a sample

The temperature measurements shall be taken at electrode positions A (temperature $T1$) and B (temperature T2) using a thermal imaging camera as shown in Figure 2. The temperature difference (ΔT=$T1$ - $T2$) is measured after reaching and stabilizing the temperature for 60 s.

The temperature T1 is the mean temperature value measured at two vertical positions at A' and A'' and T2 is the mean temperature taken at positions B' and B'' (see Figure 2).

NOTE A' and A" are positions at electrode location A. They are vertically separated as shown in Figure 2. Same for positions B' and B".



Key

1 T1 : the mean temperature value of two vertical positions at A' and A"

2 T2 : the mean temperature value of two vertical positions at B' and B"

3 length between electrodes

Figure 2 — Thermography images of carbon fibre tow measurement

8.5 Sample preparations

8.5.1 Mounting and handling

Mount the test sample on the tray and adjust the height of the cylinder until the thermal imaging camera make focus with the test sample. The distance between the thermal imaging camera and the sample shall be 200 mm to 300 mm.

8.6 No. of samples and no. of measurements

Minimum of five specimens shall be tested and at least 15 points shall be measured for each specimen.

If more than 30 points are measured consecutively for the accuracy of the measurement temperature, the test shall be stopped, and the test resumed with a minimum of 30 s rest.

8.7 Data

The data obtained from the thermal imaging camera is applied to the Fourier's thermal conductivity formula [shown as Formula (1)] for calculating the heat transfer parameter value of the 12 K carbon fibre tow.

$$K = \frac{L}{\Delta T} \times \frac{Q}{A} \tag{1}$$

where

K is the heat transfer parameter of carbon fibre in W/m·K;

L is the active fibre length in millimetres (100^{+5}_{-0} mm);

ΔT is the temperature difference T1-T2;

Q is the applied power in watts;

A is the cross-sectional area of carbon fibre tow ($4{,}89E^{-7}$ m^2).

9 Test report

The test result shall include the following information:

a) a reference to this document, i.e. ISO/TS 23483:2022;

b) description of test equipment;

c) test samples;

d) test conditions;

e) assessment items.

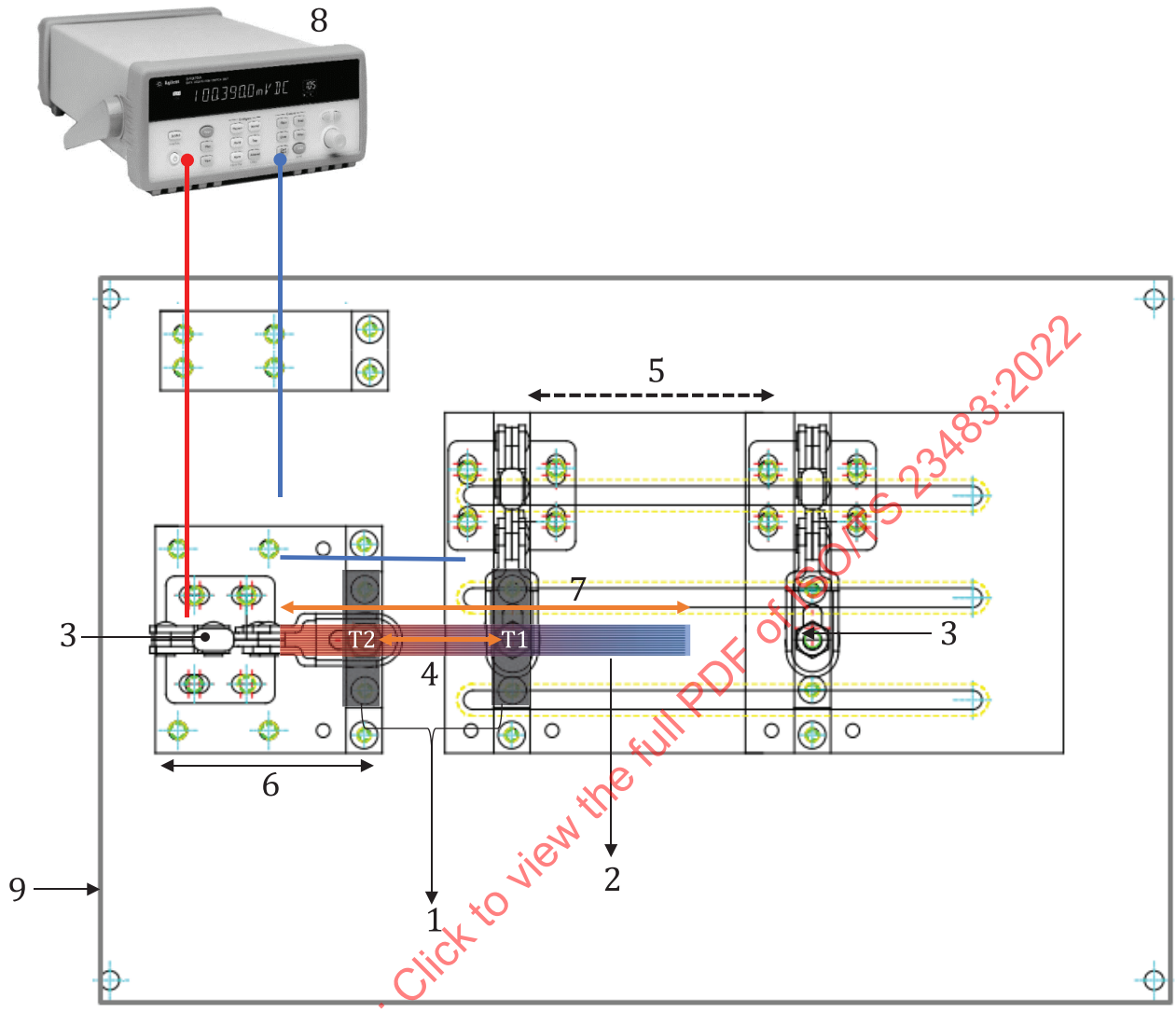
f) test results and measured values of heat transfer parameter;

g) date and time of the tests;

h) any other relevant information that may have an influence on the test results obtained.

Annex A
(informative)

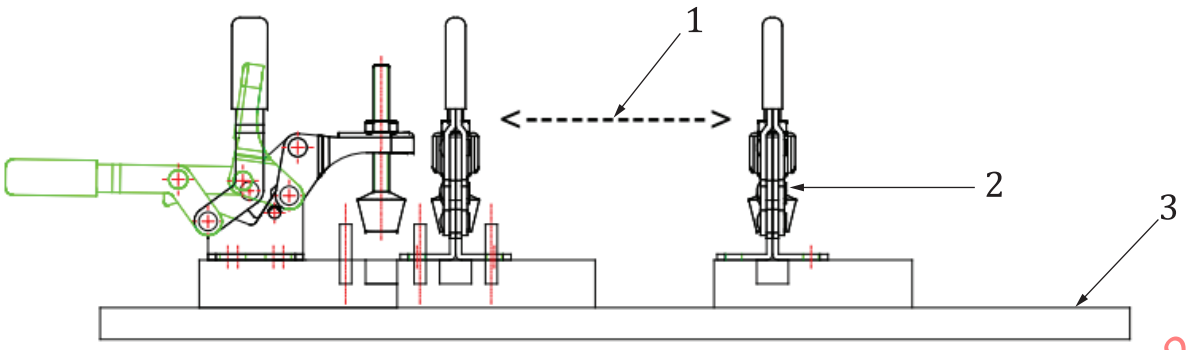
Typical set-up for heat transfer parameter measurement



Key

1 copper electrode (width: 9 mm, length: 35 mm)
2 carbon fibre tow
3 clamp
4 carbon fibre length to measure (15^{+5}_{-0} mm)
5 moving part (5 mm to 80 mm)
6 fixed part
7 total carbon fibre tow length (100^{+5}_{-0} mm)
8 DC power supply
9 plate

Figure A.1 — Example of heat transfer parameter measurement set-up for 12 K carbon fibre tow



Key

1 moving part
2 clamp
3 plate

Figure A.2 — Example of sample holder clamp side view