DRAFT FOR DEVELOPMENT

DD CEN/TS 15866:2009

Advanced technical ceramics — Ceramic composites — Determination of the thermal diffusivity of ceramic fibres

ICS 81.060.30

BSI British Standards

National foreword

This Draft for Development is the UK implementation of CEN/TS 15866:2009.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature. It should be applied on this provisional basis, so that information and experience of its practical application can be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the international organization responsible for its conversion to an international standard. A review of this publication will be initiated not later than 3 years after its publication by the international organization so that a decision can be taken on its status. Notification of the start of the review period will be made in an announcement in the appropriate issue of Update Standards.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into an international Standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent to the Secretary of the responsible BSI Technical Committee at British Standards House, 389 Chiswick High Road, London W4 4AL.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Advanced technical ceramics - Ceramic composites - Determination of the thermal diffusivity of ceramic fibres

Céramiques techniques avancées - Céramiques composites - Détermination de la diffusion thermique des fibres céramiques

Hochleistungskeramik - Keramische Verbundwerkstoffe - Bestimmung der Temperaturleitfähigkeit von keramischen Fasern

This Technical Specification (CEN/TS) was approved by CEN on 3 February 2009 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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Foreword

This document (CEN/TS 15866:2009) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

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1 Scope

This Technical Specification specifies the conditions for the determination of the thermal diffusivity of single filaments of ceramic fibres parallel to the fibre axis.

This Technical Specification applies to continuous ceramic filaments taken from tows, yarns, braids and knittings.

The experimental conditions are such that the material behaves in a homogeneous manner and that the heat transfer occurs only by thermal conduction.

The method is applicable to materials which are physically and chemically stable during the measurement, and covers the range of temperature between 100 K and 600 K. It is suitable for the measurement of thermal diffusivity values in the range between 10^{-4} m²·s^{-1} and 10^{-7} m^2·s^{-1}.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 843-5:2006, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 5: Statistical analysis*

CEN/TR 13233:2007, *Advanced technical ceramics — Notations and symbols*

EN 60584-1, *Thermocouples — Part 1: Reference tables (IEC 60584-1:1995)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

ISO 3611, *Micrometer callipers for external measurement*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TR 13233:2007 and the following apply.

3.1
thermal diffusivity
a
ratio of the thermal conductivity to the product of the bulk density and the specific heat capacity

3.2
transient half time
$t_{½}$
time from the initiation of the pulse until the increase of the temperature on the back face of the test specimen reaches one half of the maximum temperature increase

3.3
thickness
h
dimension of the test specimen in the direction of heat transfer measurement

4 Principle

One side of a plane and parallel test specimen is exposed to a uniformly distributed energy pulse that is of very short duration compared to the transient half time.

The transient temperature rise, ΔT, on the opposite face (back face) or a quantity directly proportional to ΔT is recorded as a function of time, t (see Figure 1).

The thermal diffusivity is obtained by comparing the experimental thermogram with a theoretical model, which is a uni-dimensional analytical thermal model, with two parameters, as described in Annex A. If other models are used, they are to be specified in the test report.

5 Apparatus

5.1 Heat pulse source

The heat pulse source may be a flash tube or a pulse laser. The pulse energy shall be as uniform as possible over the front face of the test specimen.

5.2 Test chamber

The test chamber shall be a furnace or a cryostat, capable of operation within the temperature range required, or a draught proof enclosure for ambient temperature measurement.

The design of the furnace shall meet the following requirements:

a) homogeneous temperature on the test piece;

b) in steady state conditions, the drift in temperature shall be less than 0,01 K/s;

c) the heat pulse source may be placed either inside the furnace or outside the furnace. In that case, the furnace shall be fitted with a window, transparent to the pulse radiation;

d) the furnace shall contain a working area in which the spatial temperature gradient is sufficiently low ($\leq$ 5 K) to ensure a consistent temperature across the sample. In addition, it shall provide suitable access for measurement of ΔT or a quantity directly proportional to ΔT on the back face of the test piece.

NOTE 1 Measurement under vacuum reduces convection losses.

NOTE 2 When the test is performed under gas, the test piece should be in a horizontal position in order to reduce convection effects of the gas on the specimen.

5.3 Detectors

5.3.1 Measurement of absolute temperature

The temperature of the test piece shall be measured either with a thermocouple, in accordance with EN 60584-1, or with an optical pyrometer.

5.3.2 Transient detectors

The detector shall be either an infrared detector or a thermocouple or any other means that does not disturb the measurement of the transient response of the specimen. It shall be capable of detecting changes of 0,05 K in the temperature of the test piece, with a linear response over the range of temperature change less than or equal to 5 K.

It shall have a response time calculated as follows:

$$t_d \leq 0{,}002\ \frac{h^2}{a} \qquad (1)$$

where

t_d is the response time, in second (s);

h is the thickness, in metre (m);

a is the thermal diffusivity, in square metre per second ($m^2.s^{-1}$).

This condition shall be verified afterwards and if it is not met, the size of the specimen shall be increased.

The infrared detector, when used, shall be of a type appropriate to the minimum test piece temperature, for example:

a) Hg/Cd/Te cell, liquid nitrogen cooled, for test specimen temperatures within the range 300 K to 800 K;

b) PbS cell for test specimen temperatures above 500 K.

Care shall be taken that the signal comes only from the central area of the back face, that is with a tolerance of 5 % of the diameter of the test specimen.

Thermocouples, when used, shall be of the separated junction type, the hot junction being the back face of the test piece. They shall be in accordance with EN 60584-1. Electrically non-conductive material shall be coated on the front face and on the rear face, with a thin coating of high thermal conductivity material in order to ensure accurate measurement of surface temperatures.

In order to minimize heat losses, the use of the thermocouples with wires of the smallest possible diameter is recommended.

NOTE The thermocouple type most often used is chromel-alumel for measurements from room temperature up to 1 100 K. Semi-conducting couples may also be used: Bi_2Te_3 from 90 K to 400 K.

5.4 Data acquisition

The data acquisition system used may be analogue or digital. It shall be equipped with means of recording the temperature change versus time (before, during and after the pulse) and the time origin. These means shall be accurate to within 0,02 ms.

6 Test specimens

The size of the test specimens shall be fixed to meet the requirements for application of the chosen thermal model (for example like those described in Annex B). Generally, a disc-shape test specimen with a diameter between 8 mm and 25 mm is used. The thickness of the specimen shall be sufficient in order to avoid influence of material homogeneity. This shall be ensured by performing tests on two series of test specimens with a thickness ratio of about 2. Recommended starting thickness are between 1 mm and 10 mm. Homogeneous material behaviour can be assumed when the mean values of the thermal diffusivity determined from each series do not differ by more than 10 %.

7 Test specimen preparation

7.1 Machining and preparation

Test specimens made from fibres shall be cut with their longitudinal axis coinciding with the fibre axis. A suggested method to prepare such test specimens is described in Annex B. It essentially consists of introducing fibres with a needle in a thermoplastic tube (as for example those used to protect electric cables). Once the required volume of fibres is reached, the plastic tube is heated and then shrinks, thereby exerting a sufficient pressure on the packed fibres to obtain a good degree of compaction as shown in the figures of Annex B. Disks can be cut to the desired thickness.

The faces shall be flat and parallel. The plan parallelism of the two faces shall be better than 0,05 mm.

If the test specimen is transparent to the infra red radiation at the considered wavelength of the laser, a coating is necessary. This coating shall be opaque, absorbent, adherent and compatible with the test specimen.

If the test specimen is non conductive and if a thermocouple is used to measure the temperature on the back face, an adequate conductive coating shall be used.

7.2 Number of test specimens

A minimum of three test specimens shall be tested at each test temperature.

If a statistical evaluation is required, the number of test specimens shall be in accordance with EN 843-5:2006.

8 Procedure

8.1 Calibration of apparatus

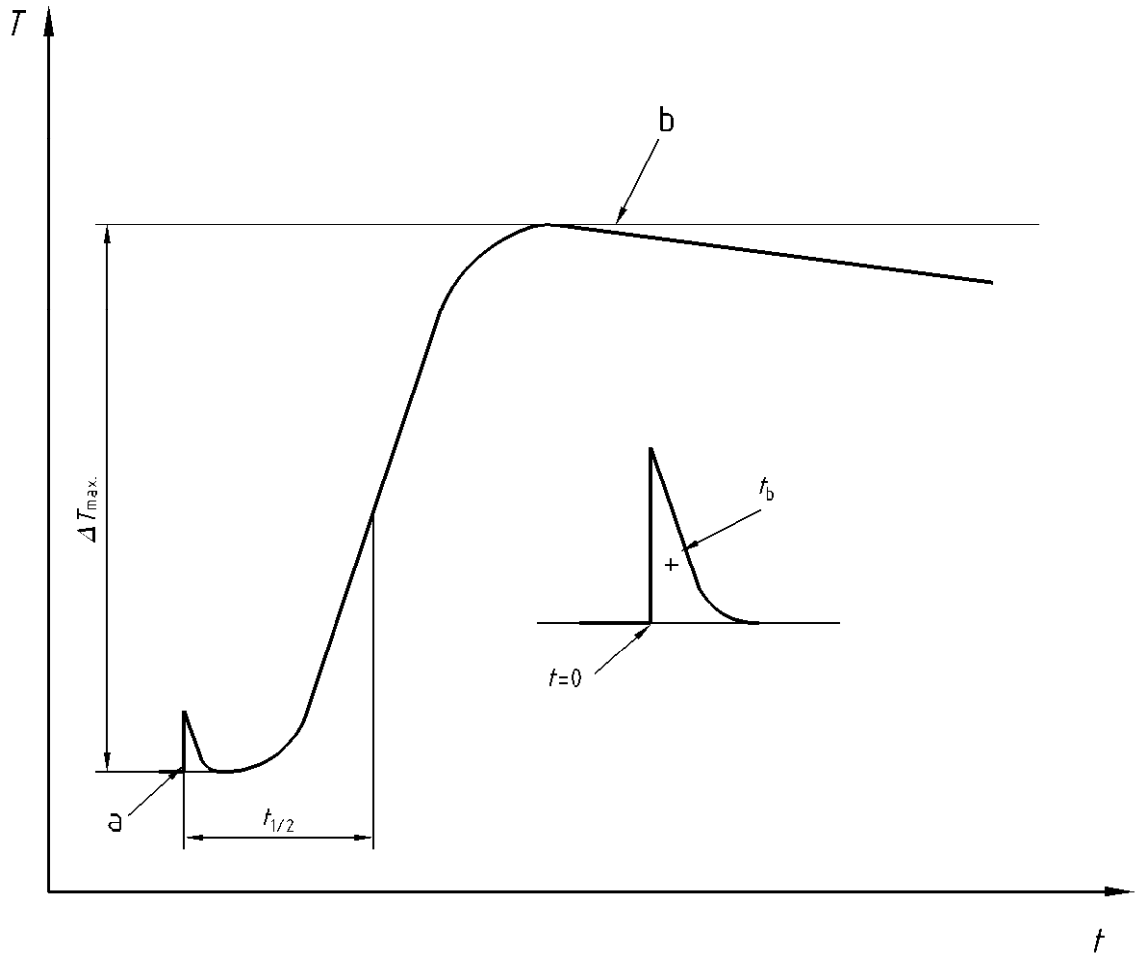
Although the measurement of thermal diffusivity is an absolute method, reference type materials with known diffusivities can be used to check the system. The homogeneity of the laser beam can be verified by photographic paper (Polaroid type).

NOTE There is no recognized standard reference material for thermal diffusivity measurements, although several materials are used (for example POCO graphite, ARMCO iron).

8.2 Test procedure

8.2.1 The pulse duration shall be less than or equal to $0{,}003\,\frac{h^2}{a}$ to allow for direct application of the theoretical model. In general, this corresponds to a period less than 1/50 of transient half time ($t_{1/2}$).

NOTE When this condition is not met, a correction of the thermogram is possible by placing the time origin at the energetic barycentre t_b of pulse (see Figure 1).



Key

T temperature

t time

a pulse triggering

b no heat losses

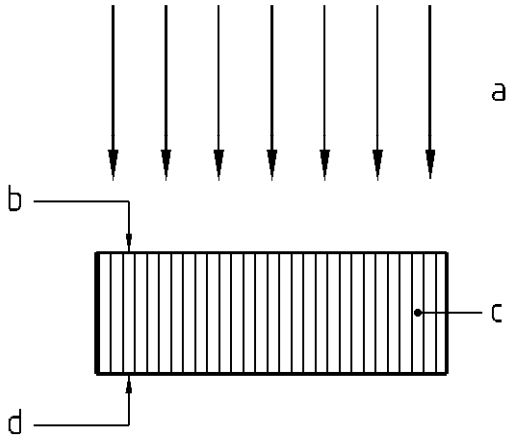
Figure 1 — Schematic representation of the temperature rise of the back face of the test specimen

8.2.2 Measure the thickness of the test specimen to the nearest 0,01 mm, using micrometer callipers in accordance with ISO 3611. In cases where a coating is used, make the measurement before coating. When the change in thickness due to thermal expansion is larger than 1 %, apply a correction to the measured thickness value.

8.2.3 Install the test specimen, such that the front face is perpendicular to the heat source beam. Thermal losses from the specimen to the surrounding environment shall be kept to a minimum and the contact area of test piece with the sample holder shall be as small as possible.

8.2.4 After the test specimen has reached constant temperature, its front face is exposed to the heat pulse and the temperature change is measured on the back face. The record shall start before the pulse in order to determine the baseline. Care should be taken to avoid possible base line shifts caused by the pulse.

8.2.5 The energy level of the heat pulse source shall produce a rise in temperature not exceeding 5 K on the back face of the test piece. If it is not the case, a new test shall be realized with a lower energy until this condition is met.



Key

a energy from pulse source
b front face
c test specimen
d back face

Figure 2 — Test configuration

9 Test validity

The following circumstances invalidate a test:

— failure to specify and record test conditions;

— temperature rise of the back face higher than 5 K.

10 Results

The value of the thermal diffusivity is determined by comparing the experimental thermogram (see Figure 1) with a set of calculated thermograms obtained by the application of a thermal model.

A number of simplifying approximations lies at the basis of the considered thermal model. These approximations impose some limitations to the range of applicability of the model and consequently some boundary conditions on the validity of the experiment. See for example Annex A.

The evaluation of the temperature rise on the back face is also possible in accordance with other thermal models which are derived from the Fourier equation of heat transfer. For every thermal model which is used, the range of application shall be considered. This range of application depends on the approximation which is made within the solution of the Fourier equation (see [1] to [6] in Bibliography).

11 Test report

The test report shall be in accordance with the reporting provisions of EN ISO/IEC 17025 and shall contain at least the following information:

a) name and address of the testing establishment;

b) date of test;

c) on each page, a unique report identification and page number;

d) customer name and address;

e) reference to this Technical Specification, i.e. determined in accordance with CEN/TS 15866;

f) an authorizing signature;

g) any deviation from the method described, with appropriate validation, i.e. demonstrated to be acceptable to the parties involved;

h) description of the test specimen: material type, manufacturing code, batch number;

i) description of the equipment used;

j) calibration procedures if applicable;

k) methods of manufacturing of the test specimens from supplied material, test specimen thickness, thickness and type of coatings (if applicable);

l) transient detector employed;

m) environmental conditions, i.e. vacuum, inert gas, etc.;

n) a statement regarding the thermal expansion of the test specimen and whether or not a correction to the thickness was applied;

o) the thermal model used;

p) individual values and average value of the thermal diffusivity;

q) number of tests carried out and the number of valid results obtained.

Annex A
(informative)

Uni-dimensional thermal model

The model makes use of two parameters, the characteristic time and Biot number. Both parameters depend on the size and thermal properties of the test specimen

The characteristic time is defined as follows:

$$t_c = \frac{h^2}{a} \tag{A.1}$$

where

t_c is the characteristic time;

h is the thickness of the test specimen;

a is the thermal diffusivity (to be determined).

The Biot number corresponding to the test piece is defined by the following equation:

$$B_i = \frac{k\ h}{\lambda} \tag{A.2}$$

where

k is the heat exchange coefficient between the test specimen and its surrounding, in Watt per square metre and per Kelvin ($W.m^{-2}.K^{-1}$);

λ is the thermal conductivity, in Watt per metre and per Kelvin ($W.m^{-1}.K^{-1}$).

$$\frac{\Delta T}{(\frac{Q}{\rho\ C\ h})} = 2\sum_{n=1}^{\infty} \frac{\mu_n^2 \left(\cos \mu_n + \frac{B_i}{\mu_n} \sin \mu_n \right)}{\mu_n^2 + 2B_i + B_i^2}\ \exp\left(-\mu_n^2\ \frac{a\,t}{h^2} \right) \tag{A.3}$$

where

ΔT is the temperature rise, in Kelvin (K);

Q is the energy absorbed per surface unit, in Joule by square metre ($J.m^{-2}$);

t is the time, in second (s);

ρ is the bulk density, in kilogram per cubic metre ($kg.m^{-3}$);

C is the specific heat capacity, in Joule per kilogram and per Kelvin ($J.kg^{-1}.K^{-1}$).

The coefficients μ_n appearing in Equation (A.3) are the roots of the following transcendental equation:

$$2\ \mu_n\ B_i\ = (\mu_n^2\ -\ B_i^2)\ \tan \mu_n \qquad \text{(A.4)}$$

This transcendental equation results from the solution of a set of differential equations that describe the heat transfer according to the unidimensional model. The roots μ_n are the eigenvalues and each of the terms in Equation (A.3) is an eigenfunction of the thermal problem.

From the above equation, it can be observed that the values of μ_n depend on the value of the Biot number B_i. Also, the argument of the exponential factor in the series equation contains the inverse of the characteristic time. It thus appears that the theoretical thermogram depends on both the Biot number and the characteristic time. Consequently, by varying the values of both parameters, two families of thermograms can be obtained. Comparison of the theoretical curve that best fits the experimental thermogram allows to determine the numerical values of the characteristic time and of the Biot number of the test specimen. From the value of the characteristic time, the value of the thermal diffusivity is obtained directly.

A number of methods exist that allow the identification of the values of the two parameters that best fit the experimental thermogram. It is recommended that the agreement obtained by these methods is checked by plotting the reduced error (the reduced error should be properly defined here) between the experimental and the best fit theoretical thermogram as a function of time. The time dependence of the reduced error should be random. Residuals with non-random time dependence shall be rejected in the evaluation of the thermal diffusivity.

A suggested identification method is the so-called “temporal moment method”. This method makes use of two moments of the thermogram, M_{-1} and M_0. These moments are defined as follows:

$$M_0 = \int_{t_{0,1}}^{t_{0,8}} \Delta T(t)\, dt \qquad \text{(A.5)}$$

$$M_{-1} = \int_{t_{0,1}}^{t_{0,8}} \frac{\Delta T(t)}{t}\, dt \qquad \text{(A.6)}$$

where

$\Delta T(t)$ is obtained from the experimental thermogram;

$t_{0,1}$ is the time needed to reach 0,1 ΔT_{max};

$t_{0,8}$ is the time needed to reach 0,8 ΔT_{max}.

The characteristic time t_c according to this method is approximated by the ratio $\frac{M_0}{F\ M_{-1}}$ where the identification function F is defined as follows:

$$F = 0{,}08548 - 0{,}32601\,(0{,}5486 - M_{-1}) + 0{,}29592\,(0{,}5486 - M_{-1})^{\,2{,}1607} \qquad \text{(A.7)}$$

Annex B
(informative)

Methods 1 and 2

B.1 Method 1

Method 1 is depicted in figures B.1 to B.3.

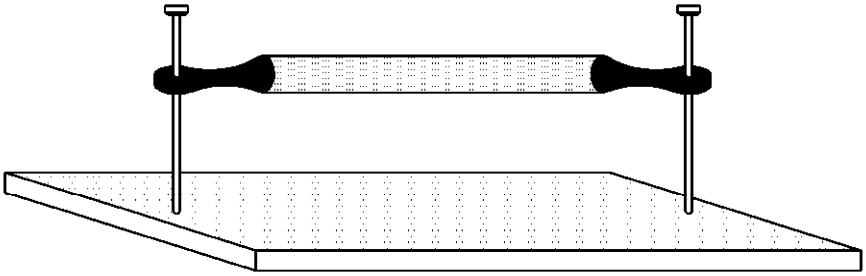
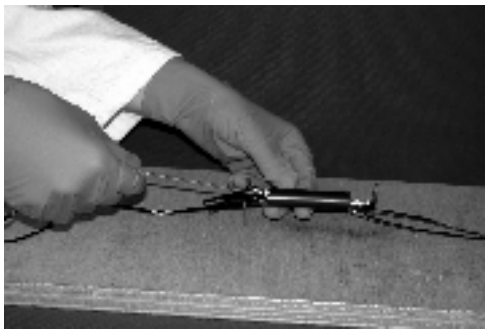
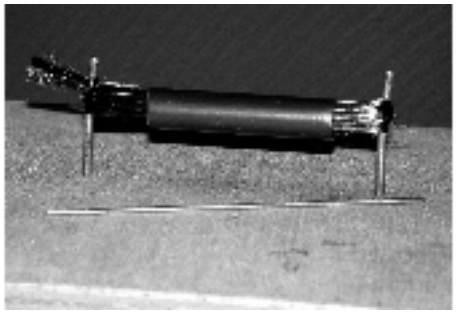


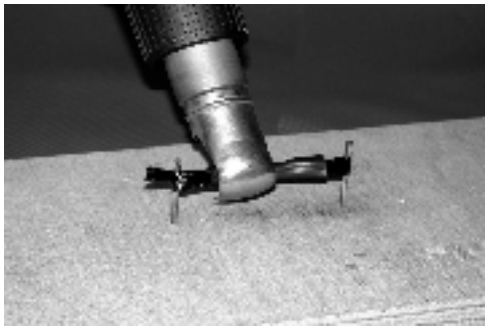
Figure B.1 — Schematic view of the test specimen preparation, showing the stretched fibre bundle in the plastic tube



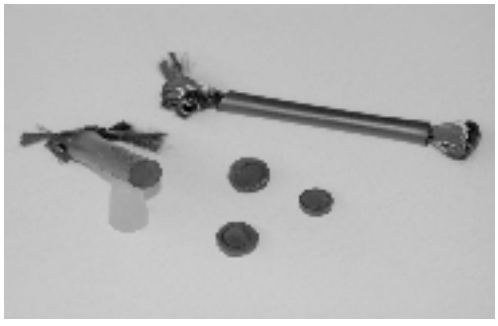
a) Insertion of the fibre bundle into the plastic tube



b) Stretched fibre bundle before heating

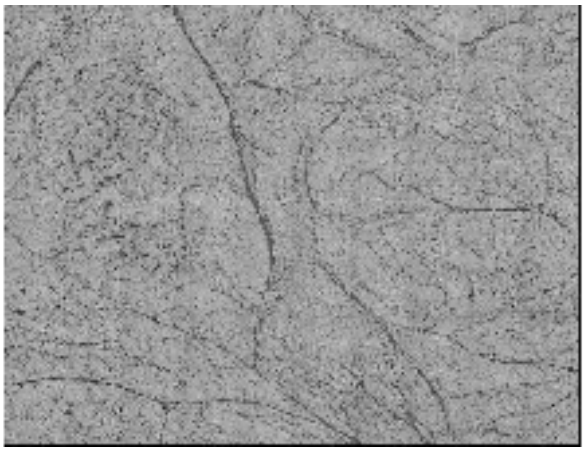


c) Heating of the plastic tube



d) After shrinkage due to heating, small disks may be cut

Figure B.2 — Preparation of the test specimen



NOTE The fibre ratio is 71 %.

Figure B.3 — Optical microscope view of the cross section of a sample, showing the high compaction ratio

B.2 Method 2

The concept of Method 2 is reasonably well-known and is shown schematically in Figure B.4.

Short lengths of fibre tow are loaded into the lower half of the compression jig until the jig is full (Figure B.5 a).

Semicircular rings are placed in the recesses in the bottom and top parts (Figure B.5 b) and the top part is forced downwards to compress the tows (Figure B.5 c).

A complete ring is slid over the two half rings to clamp the bundle (Figure B.5 c), and the assembly is removed from the jig.

Using a razor blade, the excess fibres are sliced off to leave a planar disc test-piece (Figure B.5 d).

The principal problem with this arrangement is knowing exactly how many strips of tow to place in the jig before attempting the compression. If insufficient strips are used, the bundle is too loose and the fibres are likely to move during cutting or during subsequent measurement. If too many tow strips are used, the force required is too high, and damage to fibres may occur, even if some strips are subsequently removed. The technique also employs a large quantity of tow, because there is a high wastage level in using lengths which can be handled, and the technique is tedious to employ.

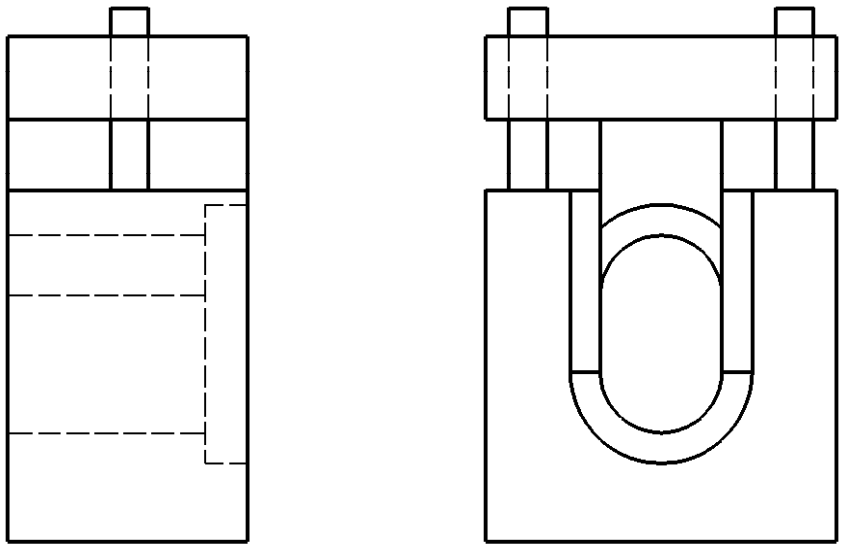
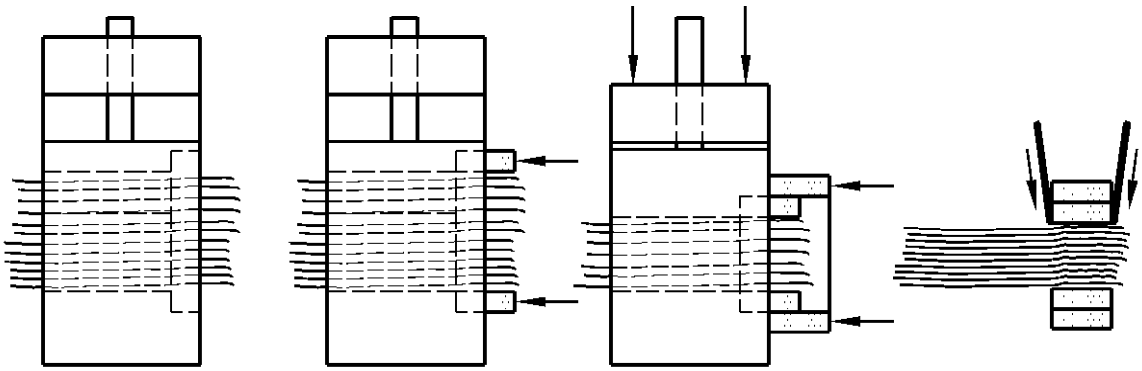


Figure B.4 — Schematic of the fibre-bundle press system for creating thermal diffusivity test-pieces



a) inserting fibre tows

b) inserting two semicircular rings

c) compressing the jig to make the half-rings meet and sliding a complete ring over them

d) removing the bundle, aligning the ring with the test-piece

Figure B.5 — Method of manufacturing test-pieces using the compression jig

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