INTERNATIONAL STANDARD

ISO 19618

First edition
2017-02

Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method for normal spectral emissivity using blackbody reference with an FTIR spectrometer

Céramiques techniques — Méthode de mesure de l'émissivité spectrale normale utilisant un corps noir de référence avec un spectromètre FTIR

Reference number
ISO 19618:2017(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 on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement method for normal spectral emissivity using blackbody reference with an FTIR spectrometer

1 Scope

This document specifies a method used for the determination of normal spectral emissivity and normal quasi-total emissivity of fine ceramics using blackbody reference with a Fourier transform infrared spectrometer (FTIR) at elevated temperatures. This method is applicable to fine ceramics, ceramic matrix composites, and continuous fibre-reinforced ceramic matrix composites which are opaque and highly non-reflective at wavelengths between 1,67 µm and 25 µm. The applicable temperature range is approximately 350 K to 1 100 K.

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.

IEC 60584-2, *Thermocouples — Part 2: Tolerances*

IEC 60751, *Industrial platinum resistance thermometers and platinum temperature sensors*

3 Terms, definitions and symbols

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:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
emissivity
ε
ratio of the radiant emittance of a substance (specimen) to the radiant emittance of a *blackbody* (3.2) at the same temperature

3.2
blackbody
ideal thermal radiator that absorbs all incident radiation completely, whatever the wavelength, direction of incidence or polarization

3.3
spectral emissivity
$\varepsilon_S\,(\lambda, T)$
emissivity (3.1) of a specimen at a defined wavelength λ and temperature T

3.4
normal spectral emissivity
$\varepsilon_{ns}(\lambda, T)$
emissivity (3.1) perpendicular to the specimen at a defined wavelength λ and temperature T

3.5
normal total emissivity
$\varepsilon_{n}(T)$
ratio of the normal component of the total emissive power of a specimen surface to the normal component of the total emissive power of a blackbody at the same temperature T

3.6
normal quasi-total emissivity
$\varepsilon_{n}(\lambda_1, \lambda_2, T)$
normal emissivity between λ_1 and λ_2 at temperature T

Note 1 to entry: Calculated as the ratio of the normal component of the emissive intensity of a specimen between λ_1 and λ_2 to the normal component of the emissive intensity of a blackbody between λ_1 and λ_2 at the same temperature T.

4 Principle

The infrared radiance spectrum data from a specimen surface and from a blackbody furnace are measured using an FTIR spectrometer. The normal spectrum emissivity of a specimen is determined by direct comparison to a blackbody reference data at the same temperature.

Integrating the infrared radiance spectrum data in the specified wavelength range numerically, normal quasi-total emissivity is calculated.

5 Apparatus

5.1 Measurement system

The measurement system consists of a Fourier transform infrared spectrometer (FTIR), specimen heating device, blackbody furnace, and temperature measuring devices as shown in Figure 1.

5.2 Fourier transform infrared spectrometer (FTIR)

Infrared radiation from a specimen or a blackbody furnace is let into a Mickelson interferometer of an FTIR through an external optical path. Thereby, an interferogram of infrared radiation is obtained. The infrared radiance spectrum is obtained numerically by Fourier transformation processing from the interferogram.

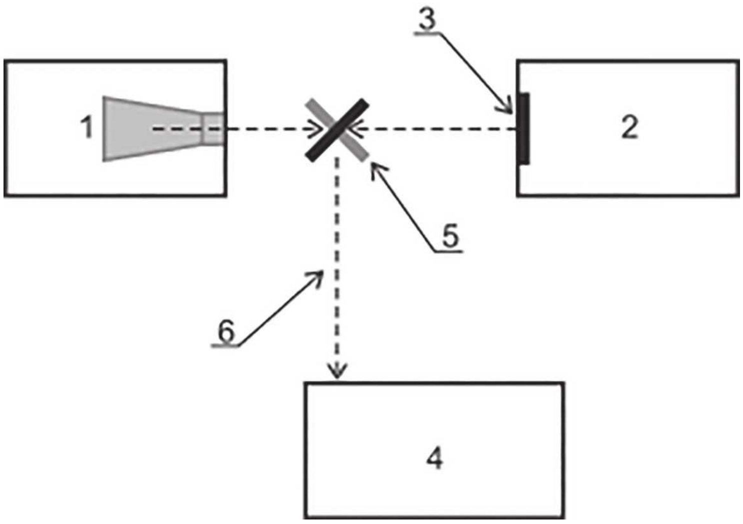
The optical system including a Mickelson interferometer of an FTIR shall be filled with dry N_2 or dry air of which the dew point is lower than 220 K to reduce the effect of H_2O and CO_2 in air. Vacuum may be used.

The measurement spot area at the sample position and at the blackbody furnace positions shall be measured preliminarily.

5.3 Specimen heating device

A specimen shall be heated using a heating device such as electrical resistance heating elements, heat-pipes, heat-transfer media, etc. The specimen surface temperature shall be well controlled to within ± 3 K.

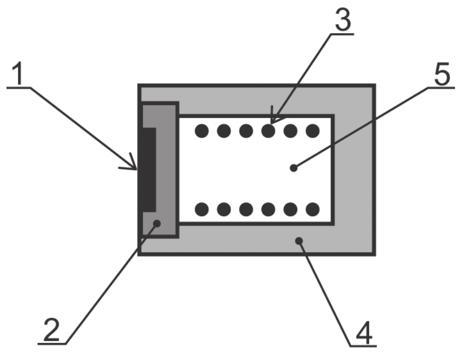
An example of a specimen heating device is depicted in Figures 2 and 3.



Key

1 blackbody furnace
2 specimen heating device
3 specimen
4 FTIR spectrometer
5 switchable mirror
6 external optical path

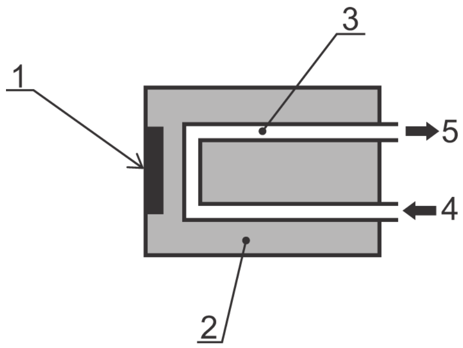
Figure 1 — Measurement system



Key

1 specimen
2 hot plate (block)
3 resistance heater elements
4 thermal insulator
5 cavity

Figure 2 — Example of a specimen heating device: electrical resistance furnace



Key

1 specimen

2 device body

3 channel for heat transfer media

4 inlet

5 outlet

Figure 3 — Example of a specimen heating device: heat transfer media

5.4 Blackbody furnace

Total emissivity of a blackbody furnace shall be higher than 0,95 and shall be internationally traceable. The aperture size shall be greater than three times the measurement spot area of an FTIR.

5.5 Temperature measuring devices and thermometer

All temperature measuring devices (temperature sensor) shall be internationally traceable.

The temperature sensor used for a test specimen shall be thermocouple in accordance with IEC 60584-2, or resistance temperature detector in accordance with IEC 60751.

The sensor diameter shall be as small as possible to prevent heat transfer through the sensor wires.

5.6 Mirror

A mirror with reflection index of more than 0,95 is used to measure background infrared radiance spectrum at room temperature. A gold-coated mirror should be used.

6 Test specimens

A material shall be opaque and highly non-reflective at wavelengths between 1,67 μm and 25 μm.

— The specimen shall have plain plate geometry. The specimen area shall be more than three times the measurement spot area of a spectrometer at a measurement position. Typical dimensions are 2 mm to 6 mm thick, and 10 mm to 50 mm in diameter or in square.

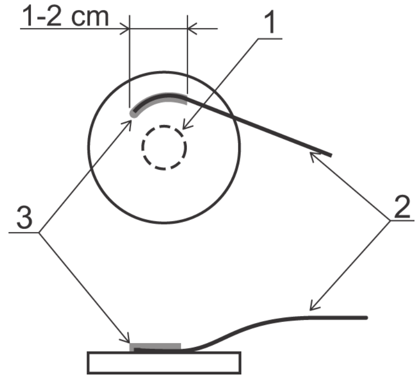
— The back side of a specimen shall be flat and smooth to be in close contact with a specimen heating device.

— The machining parameters for specimen surfaces should be documented.

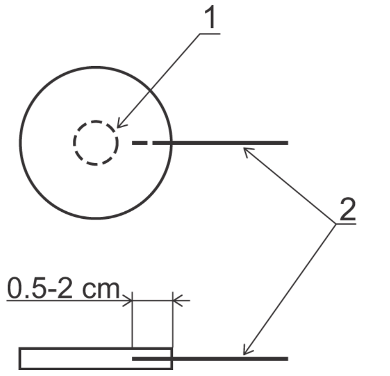
A temperature sensor such as a thermocouple shall be contacted firmly to a specimen. The two methods shown in Figure 4 should be used.

— A temperature sensor is bonded on a specimen surface using welding or adhesion. The temperature sensor and adhesive shall not appear inside of the measurement spot area. A few centimetres of the temperature sensor should be bonded on the surface to prevent thermal conduction through the sensor wires.

— A temperature sensor is embedded into a small hole machined from a specimen side surface. The embedded length should be more than 5 mm to 20 mm to prevent thermal conduction through the sensor wires.



a) Bonded on a specimen using adhesive



b) Embedded in a specimen from side surface

Key

1 measurement spot area

2 temperature sensor

3 adhesive

Figure 4 — Temperature sensor attachment methods

7 Measurement preparation

7.1 Position of a blackbody furnace and a specimen

A specimen and a blackbody furnace shall be placed at the optically equivalent position from an FTIR spectrometer.

A measurement spot area shall be at the centre of an aperture of the blackbody furnace and at the centre of a specimen.

7.2 Wavelength calibration

The spectral resolution shall be better than 16 cm^{-1}. The number of data acquisitions and integrations shall be greater than 100. Arbitrary data smoothing methods are applicable.

Wavelength (wavenumber) calibration of an FTIR shall be conducted using a polystyrene calibration film. The test results shall be documented.

7.3 Verification of linearity

The linearity of infrared radiance spectrum data shall be verified before measurement. The following two methods should be used.

a) The infrared radiance spectrum of a blackbody furnace, $[I_b(\lambda,T)]$, is measured at three different temperatures ($T_1 < T_2 < T_3$). The ratios of the measured spectrum, $I_b(\lambda,T_1)/I_b(\lambda,T_3)$ and $I_b(\lambda,T_2)/I_b(\lambda,T_3)$, are calculated. The results are shown in a graph as a function of wavelength. The theoretical infrared radiance spectrum at each temperature, $L(\lambda,T)$, is calculated using Planck's blackbody radiation function (see Annex A). The ratios $L(\lambda,T_1)/L(\lambda,T_3)$ and $L(\lambda,T_2)/L(\lambda,T_3)$ are calculated. The theoretical curves are superimposed on the graph. Comparing the measured data and the theoretical curves, the linearity of an FTIR system shall be verified. The test results shall be documented.

b) The infrared radiance spectrum of a blackbody furnace is measured at the test temperature. A 50 % infrared neutral density filter is inserted in the external optical system; then the infrared radiance spectrum of a blackbody furnace is measured. Furthermore, cutting off an external optical system, the infrared radiance spectrum is measured. By comparing the measured data (100 %, 50 %, and 0 %), the linearity of an FTIR system shall be verified. Test results shall be documented.

7.4 Verification of stability

The stability of measurement systems shall be verified by the measurement of infrared radiance spectrum from a blackbody furnace. The infrared radiance spectrum from a blackbody is measured twice within 10 min. The ratio of these two results shall be within (100 ± 1) %. The test results shall be documented.

7.5 Validation of measurement system

The measurement system shall be validated by comparing the measured data and literature data of some standard materials. The measurement procedure shall be in accordance with Clause 9.

Alumina, partially stabilized zirconia, β-silicon carbide, β-silicon nitride, glassy carbon, and graphite are often used as standard materials. The test results shall be documented.

The Christiansen wavelength can be useful to validate the measurement system, see Annex B.

8 Test condition

The room temperature shall be (23 ± 5)°C. Relative humidity should be less than 60 %. The air stream from an air conditioning system shall not flow directly on a specimen.

9 Test procedure

9.1 Background infrared radiance spectrum measurement

A gold-coated mirror is placed within ± 5 mm of a specimen position at room temperature. The normal direction of the mirror surface is inclined at a 10° angle to the optical axis of an FTIR to prevent direct reflection to the FTIR itself.

Background infrared radiance spectrum is measured at least twice within 10 min. The ratio of these results shall be within (100 ± 1) %. One experimentally obtained result among them is used as a background infrared radiance spectrum.

9.2 Specimen installation

A specimen is installed carefully on a specimen heating device with no gap separating the specimen and the heating device. A measurement spot area shall be at the centre of a specimen.

The normal direction of a specimen surface is inclined at a 10° angle to the optical axis of an FTIR to prevent direct reflection of the infrared radiance to the FTIR itself.

A temperature sensor attached to a specimen is connected to a digital thermometer. The measurement value is checked.

9.3 Infrared radiance spectrum measurement

The test procedure as the follows.

— Both a blackbody furnace and a specimen are heated to the same temperature. After reaching the test temperature, both are kept at that temperature for more than 10 min.

— Infrared radiance spectrum from a blackbody furnace is measured at least twice within 10 min. The ratio of these results shall be within (100 ± 1) %. One experimentally obtained result among them or numerical average result is used as a background infrared radiance spectrum.

— The infrared radiance spectrum from a specimen shall be measured at least twice within 10 min. The ratio of these results shall be within (100 ± 1) %. One experimentally obtained result among them or numerical average result is used as a background infrared radiance spectrum.

— All measurements taken at the same temperature shall be conducted within 30 min.

10 Calculations

10.1 Normal spectral emissivity

Calculate the normal spectral emissivity using Formula (1):

$$\varepsilon_{\mathrm{ns}}(\lambda,T)=\frac{I_{\mathrm{s}}(\lambda,T)-I_{\mathrm{m}}(\lambda,T_{\mathrm{a}})}{I_{\mathrm{b}}(\lambda,T)-I_{\mathrm{m}}(\lambda,T_{\mathrm{a}})} \tag{1}$$

where

λ	is the wavelength, in µm;
T	is the test temperature, in K;
T_a	is the room temperature, in K;
$\varepsilon_{ns}(\lambda,T)$	is the spectral emissivity at a defined wavelength λ and temperature T;
$I_s(\lambda,T)$	is the infrared radiance intensity of a specimen at a defined wavelength λ and temperature T;
$I_b(\lambda,T)$	is the infrared radiance intensity of a blackbody furnace at a defined wavelength λ and temperature T;
$I_m(\lambda,Ta)$	is the background infrared radiance intensity at a defined wavelength λ and room temperature T_a.

10.2 Normal quasi-total emissivity

Calculate the normal quasi-total emissivity using Formula (2):

$$\varepsilon_n\left(\lambda_1,\lambda_2,T\right)=\frac{\int_{\lambda_2}^{\lambda_1}\left\{I_s\left(\lambda,T\right)-I_m\left(\lambda,T_a\right)\right\}d\lambda}{\int_{\lambda_2}^{\lambda_1}\left\{I_b\left(\lambda,T\right)-I_m\left(\lambda,T_a\right)\right\}d\lambda} \qquad (2)$$

where

λ_1	is the lower limit of wavelength for the calculation, in µm;
λ_2	is the upper limit of wavelength for the calculation, in µm;
$\varepsilon_n\ (\lambda_1, \lambda_2,T)$	is the normal quasi-total emissivity between λ_1 and λ_2 at temperature T;
$I_s(\lambda,T)$	is the infrared radiance intensity of a specimen at a defined wavelength λ and temperature T;
$I_b(\lambda,T)$	is the infrared radiance intensity of a blackbody furnace at a defined wavelength λ and temperature T;
$I_m(\lambda,T_a)$	is the background infrared intensity spectrum at a defined wavelength λ and room temperature T_a.

Trapezoidal or Simpson method is often used for the numerical integration. If the integral interval is between 2,5 µm and 25 µm, normal quasi-total emissivity below 900 K is almost identical to normal total emissivity. For more information see Annex C.

11 Test report

The test report shall contain at least the following information:

a) name and address of the testing establishment;

b) date of the test, unique identification of report and of each page, customer name and address and signatory;

c) a reference to this document, i.e. “determined in accordance with ISO 19618”;

d) description of measurement system (specification of FTIR, blackbody furnace, temperature measurement device, specimen heating device, etc.);

e) description of test material and specimen (material type, manufacturing code, batch number, specimen geometry, surface finishing condition, etc.);

f) description of test set-up (resolution, number of sampling, data smoothing method, test temperature, specimen temperature measurement method);

g) normal spectral emissivity as a function of wavelength, in µm;

h) normal quasi-total emissivity;

i) validation data of testing system (wavelength (polystyrene data), linearity, stability (blackbody furnace), test results of standard materials, etc.).

Annex A
(informative)

Calculation of theoretical infrared radiance spectrum $L(\lambda,T)$ using Planck's blackbody radiation function

Planck's blackbody radiation spectrum, $L(\lambda,T)$, used in 7.3 is represented by Formula (A.1):

$$L\left(\lambda,T\right)=\frac{c_1/\pi}{\lambda^5}\left(\exp\left(\frac{c_2}{\lambda T}\right)-1\right)^{-1} \quad \text{(A.1)}$$

where

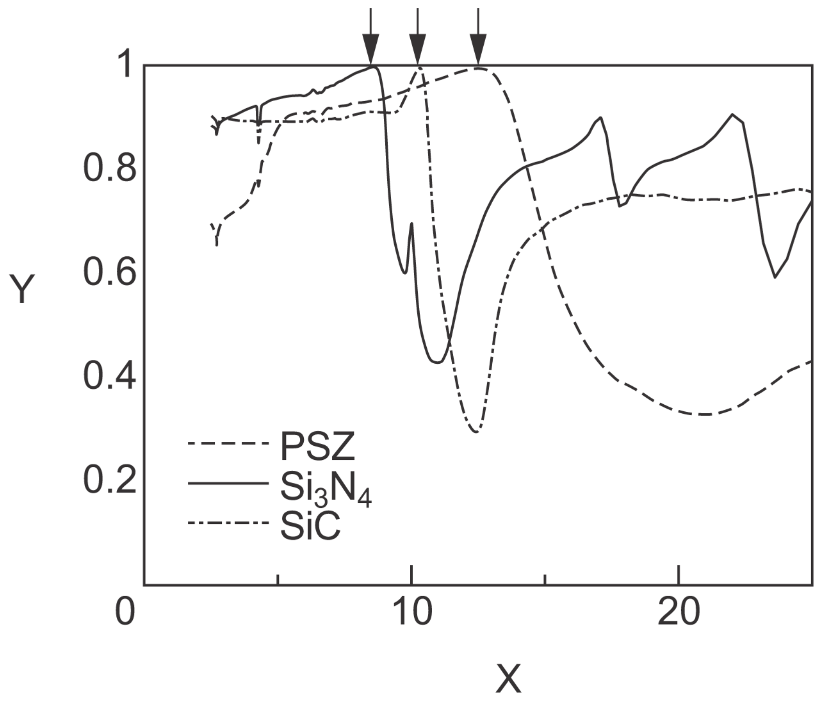
$L(\lambda,T)$	is the theoretical infrared radiance spectrum, in W/sr^{-1}/m^3;
λ	is the wavelength, in m;
c_1	is equal to 2πhc^2 (3,741 8 × 10^{-16}), in W/m^2;
c_2	is equal to hc/k (1,438 8 × 10^{-2}), in m/K;
k	is Boltzmann's constant (1,380 7 × 10^{-23}), in J/K;
h	is Planck's constant (6,626 1 × 10^{-34}), in J/s;
c	is the speed of light in vacuum (2,997 9 × 10^8), in m/s.

Annex B
(informative)

Christiansen effect

Some monolithic ceramics exhibit the Christiansen wavelength, at which the emissivity is unity[1]. Examples of normal spectrum emissivity of partial stabilized zirconia (PSZ), β-silicon carbide, and β-silicon nitride are shown in Figure B.1.

The Christiansen wavelength is useful to validate the measurement system in 7.5. If the emissivity at the Christiansen wavelength is not unity, then the optical path and the temperature of a blackbody furnace and of a specimen should be checked carefully.



Key

X wavelength, in µm

Y emissivity

PSZ partial stabilized zirconia

Si_3N_4 β-silicon nitride

SiC β-silicon carbide

NOTE Christiansen effect is observed clearly for each material.

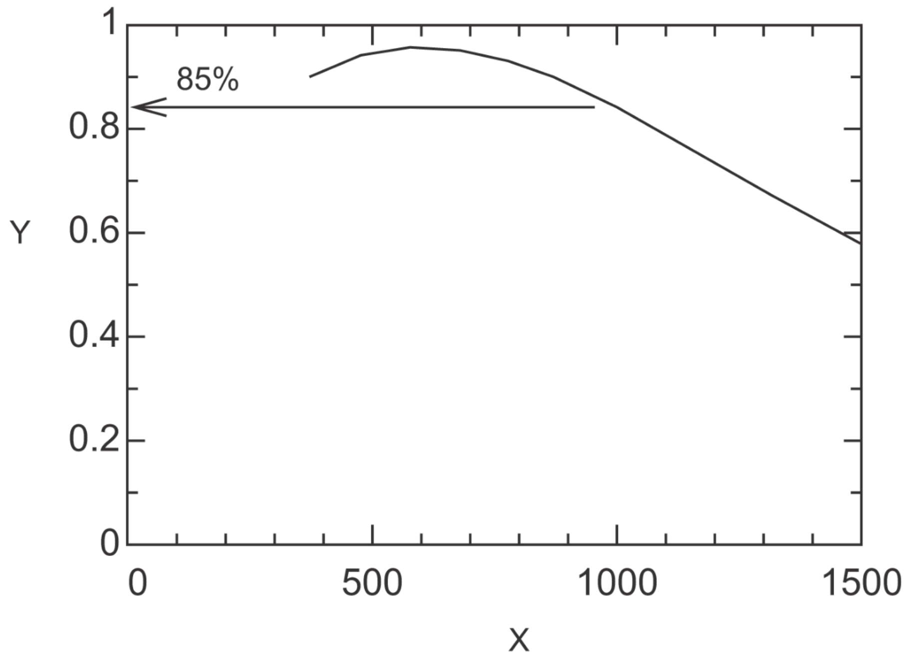
Figure B.1 — Normal spectrum emissivity of partial stabilized zirconia, β-silicon carbide and β-silicon nitride

Annex C
(informative)

Validity of normal quasi-total emissivity

Triglycine sulfate (TGS) or wide-band MCT (mercury cadmium telluride) detector is often used for FTIR spectrometry. The wavelength range is approximately 2,5 µm to 25 µm. The ratio of the emissivity between 2,5 µm and 25 µm to the total emissivity is calculated using Formula (A.1). The results are shown in Figure C.1.

The emissivity between 2,5 µm and 25 mm exceeds 85 % of the total emissivity up to 950 K. Thereby the quasi-total emissivity can be regarded as the total emissivity, as described in 10.2.



Key

X temperature, in K

Y ratio of quasi-total emissivity to total emissivity

Figure C.1 — Ratio of the quasi-total emissivity calculated between 2,5 µm and 25 µm to the total emissivity (0- ∞)

Bibliography

[1] Rousseau B., Brun J.F., De Sousa Meneses D., Echegut P. Temperature measurement: Christiansen wavelength and blackbody reference. *Int. J. Thermophys.* 2005, **26** (4) pp. 1277–1286

ICS 81.060.30
Price based on 13 pages