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Metallic and other inorganic coatings — Test method of cyclic heating for thermal-barrier coatings under temperature gradient

Revêtements métalliques et autres revêtements inorganiques — Méthode d'essai de cyclage thermique de systèmes barrière thermique sous gradient de température

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Contents

Page

Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 13123 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

Introduction

Thermal-barrier coatings (TBCs) are refractory coatings which provide thermal insulation for turbine blades and vanes, as well as for combustion chamber liners in power generation, aviation gas turbines and rocket combustors. They allow operation at substantially higher surface temperatures than is possible with bare metal, and thus TBCs have been used to extend the life of components that suffer from severe heat load cyclically during operation.

Conventional isothermal test methods are not suitable for evaluating the TBC under high heat load with a large temperature-gradient condition. Standardization of a cyclic heating test method for determination of their thermal-barrier performance and cyclic heat resistance under a temperature gradient field is required.

Metallic and other inorganic coatings — Test method of cyclic heating for thermal-barrier coatings under temperature gradient

1 Scope

This International Standard applies to the test method of cyclic heating to evaluate the thermal-barrier performance and cyclic heat resistance of the thermal-barrier coatings provided for high-temperature components, such as burners, rotor and stator blades, etc. of power-generation gas turbines used in thermal power plants, aircraft engines and rocket engines.

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.

ISO 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method*

ISO 2063, *Thermal spraying — Metallic and other inorganic coatings — Zinc, aluminium and their alloys*

ISO 2178, *Non-magnetic coatings on magnetic substrates — Measurement of coating thickness — Magnetic method*

ISO 14917, *Thermal spraying — Terminology, classification*

ISO 80000-1, *Qualities and units — Part 1: General*

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

IEC 60584-2:1982, *Thermocouples — Part 2: Tolerances*

IEC 60584-3:2007, *Thermocouples — Part 3: Extension and compensating cables —Tolerances and identification system*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2063, ISO 14917 and the following apply.

3.1
temperature gradient
temperature gradient caused by heating and cooling of both material surfaces of a test piece with thermal-barrier coatings

3.2
cyclic-heating testing
testing in which a temperature gradient is applied cyclically to the test piece with a thermal-barrier coating

3.3
number of thermal cycles
number of times the cyclic heating test is applied to the test piece with thermal-barrier coatings

3.4
acoustic emission
AE
phenomenon of a test piece provided with a thermal-barrier coating to emit an elastic wave by releasing the energy previously accumulated during damage generation like spalling, cracking, etc.

3.5
spalling area ratio
ratio of the total spalling area of a thermal-barrier coating to the effective area to which the thermal-barrier coating has been applied

4 Principle

The test piece provided with a thermal-barrier coating is subject cyclically to the cyclic heating test, in which the variation in equivalent effective thermal conductivity or acoustic emission occurrence frequency is measured, for the purpose of evaluating the thermal-barrier performance of the thermal-barrier coatings.

The test is also used to evaluate the cyclic heat resistance of thermal-barrier coatings by visually observing spalling and cracking in the test piece, and by determining the number of heating cycles needed to reach the damage tolerance limit.

5 Test piece

The typical shape and dimensions of the test piece are shown in Figure 1.

The shape and dimensions of a typical test piece shall be a disk having a diameter of 15 mm to 30 mm.

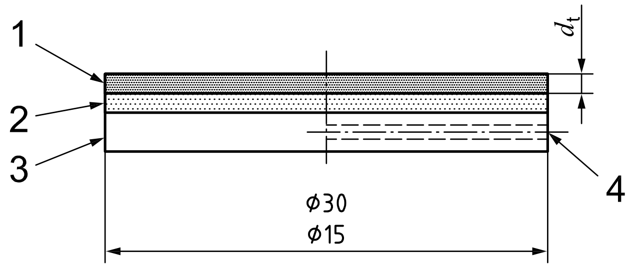
The effect of substrate thickness on the performance should be determined based on the agreement between the parties involved prior to the test. Its thickness should be measured according to ISO 1463 and ISO 2178.

It is recommended that the edge of the heating side of the substrate be rounded to a curvature of about 0,5 mm to 2 mm, or chamfered, in order to prevent cracking at the test-piece edge.

The thermal-barrier coating shall cover the entire heating surface of the substrate. The coating thickness shall be measured at multiple points in the middle and peripheral portions and shall be within ±10 % of the average value.

To measure the temperature in the central portion of the substrate, a hole may be provided for the thermocouple, running from the side of the substrate to the central portion (see 6.1.2.2). The position of the hole shall be such that no adverse effect is exerted on the stress field occurring in the coating layer and on the temperature measurement accuracy. The maximum diameter of the hole for the thermocouple should be close to the outside diameter of the thermocouple isolation. The position and diameter of the hole can be measured and described in the report, if necessary.

Dimensions in millimetres



Key

1 top coat

2 bond coat

3 substrate

4 thermocouple hole

d_t top coat thickness (mm)

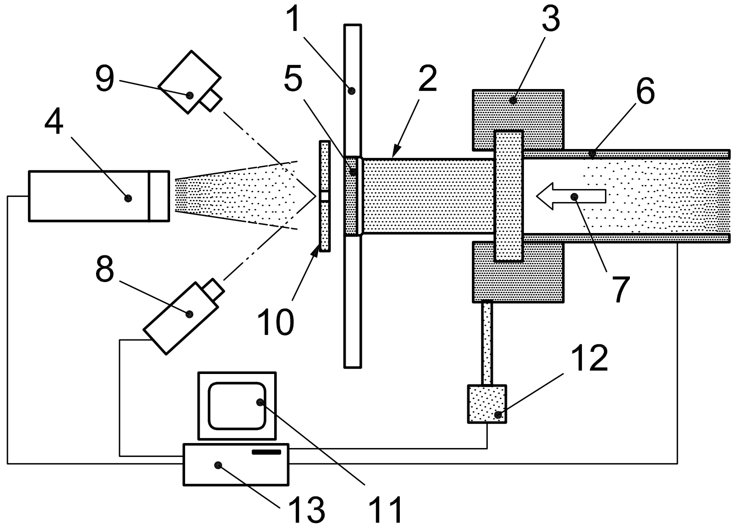
Figure 1 — Typical shape and dimensions of test piece

6 Test method

6.1 Test equipment

6.1.1 General

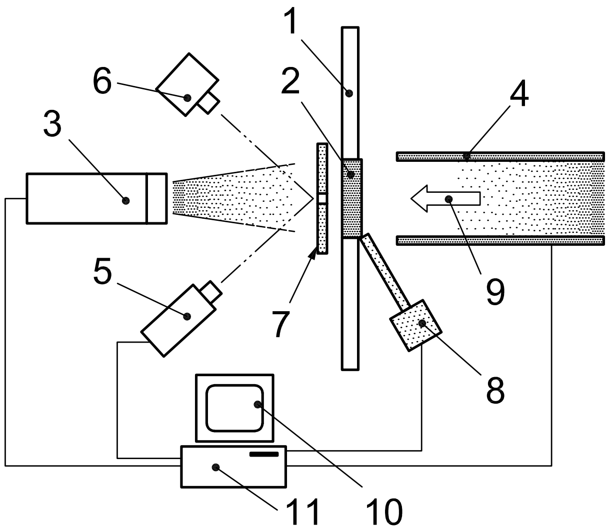
The test equipment shall consist of a heating unit, cooling unit, test block, controller, and detector/measurement instruments. Figure 2 shows a typical test equipment arrangement.



Key

1 heat shield
2 heat flux meter
3 test-piece holder
4 heating unit
5 test piece
6 cooling unit
7 coolant
8 pyrometer
9 camera
10 shutter
11 computer
12 AE sensor
13 control and detector unit

a) Test equipment with heat flux meter



Key

1 heat shield
2 test piece
3 heating unit
4 cooling unit
5 pyrometer
6 camera
7 shutter
8 AE sensor
9 coolant
10 computer
11 control and detector unit

b) Test equipment without heat flux meter

Figure 2 — Schematic diagram of test equipment

6.1.2 Test block

6.1.2.1 General

The test block shall consist of a test piece, heat flux meter and test-piece holder.

6.1.2.2 Heat flux meter

Typical examples of a heat flux meter with a test piece are shown in Figure 3.

The heat flux meter shall be brazed to the test piece as shown in Figure 3 a).

A thermal-barrier coating may be directly coated on the top of a heat flux meter as shown in Figure 3 b), in order to avoid the incomplete bonding or interdiffusion. The effect on the performance of the substrate thickness in the case of direct bonding should be considered and agreed upon between the parties involved.

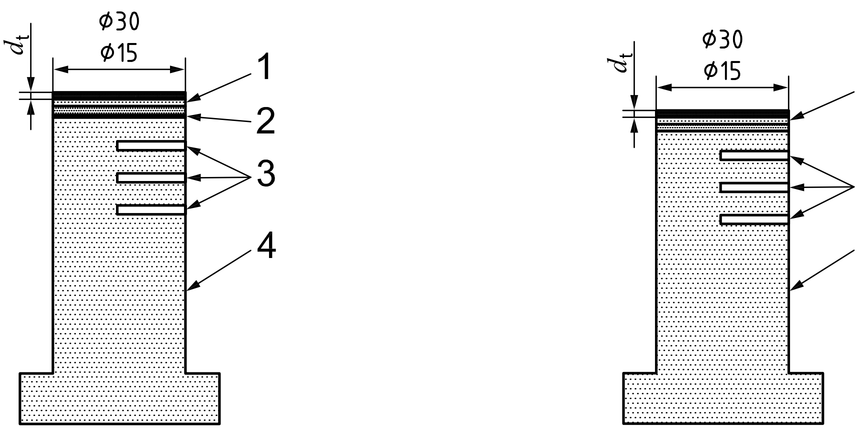
The outside diameter of the heat flux meter shall be the same as that of the test-piece substrate.

The material of the heat flux meter should have a high thermal conductivity and a small temperature dependence, normally such as copper, nickel, etc.

In order to determine the heat flux, multiple-thermocouple holes shall be provided, at regular intervals, running from the heat flux meter's outer surface through to its central axis. The number of holes and the intervals separating them shall be chosen based on the material and dimensions of the heat flux meter, test conditions, etc.

The heat flux meter need not be used when the test is intended only for evaluation of the cyclic heat resistance.

Dimensions in millimetres



a) **Brazing method**

b) **Direct coating method**

Key

1 test piece

2 thermocouple hole

3 brazing layer

4 heat flux meter

d_t top coat thickness (mm)

Figure 3 — Shape and dimensions of heat flux meter

6.1.2.3 Test-piece holder

A typical example of a test-piece holder is shown in Figure 2.

The test-piece holder is used to fix the test piece with the heat flux meter to the cooling unit.

When the AE sensor is to be used for detection of damage to the test piece, such a sensor shall be installed in the test-piece holder. The test piece may also be held by providing a notch around the test piece.

6.1.3 Heating unit

A burner, arc discharge, plasma, infrared lamp, laser beam or electron beam may be used as heating source. The source shall also have sufficient heating capacity to reach the specified temperature and shall be subject to agreement between the parties involved. The temperature of the top coat should be set to within a ±5 % range of the top coat surface temperature. The unit may be structured in such a manner as to have a shutter that is opened/closed for cyclic heating or to move the heating source for heating. A heat shield cooled with water should be set to avoid heating the side of the test piece. Insulation material may be placed around the heat flux meter to avoid radial heat loss. It is recommended that energy deposition be considered when using an e-beam or laser heating method.

6.1.4 Cooling unit

The cooling unit cools the cooling side of the test piece by means of a coolant, such as air, water, etc. This unit shall have sufficient cooling capacity to reach the required temperature gradient. The unit may also be structured to perform cooling by moving the cooling source or may have an attachment to cool the heating side.

6.1.5 Controller

The controller shall be able to control the temperature gradient and its repetition. The temperature gradient shall be reached by controlling the output of the heating unit and the coolant flow rate. Cyclic heating and cooling shall be achieved by opening/closing the shutter or by moving the heating and cooling sources.

6.1.6 Measuring instruments and sensor

6.1.6.1 General

The measuring instruments and sensor shall be as described in 6.1.6.2 to 6.1.6.5.

6.1.6.2 Thermocouple

The thermocouple used to measure the temperature distribution in the test-piece substrate and heat flux meter shall have adequate dimensions and accuracy and shall be as specified in IEC 60584. The outside diameter of the thermocouple isolation should be around 1 mm.

6.1.6.3 Non-contact-type thermometer, such as a radiation pyrometer

The radiation pyrometer used to measure the test-piece top coat surface temperature shall have an accuracy equal to or better than that of the thermocouple, within the measurement temperature range, and shall have been adequately calibrated.

6.1.6.4 Visual inspection with camera

It is recommended that visual inspection be used to detect any damage on the heated surface of the test piece. Data collection and evaluation can be automated.

6.1.6.5 Acoustic-emission-measuring unit

It is optionally recommended that the acoustic-emission-measuring unit be used to detect any damage occurring in the test piece (see 6.4). The frequency response performance of the acoustic emission transducer shall be 100 Hz to 1 MHz, the gain of the pre-amplifier shall be 40 dB or more, the gain of the main amplifier shall be 25 dB or more, and the band-pass filter shall be used as required.

6.1.7 Chamber

A chamber shall be provided around the testing block when performing the test, e.g. in the reduction atmosphere.

6.2 Testing

6.2.1 General

The testing conditions shall be agreed upon between the parties involved prior to the test.

6.2.2 Procedure

The cyclic heating test shall be carried out according to the procedure described below.

a) Activate the cooling source and confirm that the cooling surface temperature of the test piece is stable.

b) Activate the heating source and confirm that the source is stable at the required output.

c) Start temperature measurement and AE measurement.

d) Heat the heating surface of the test piece by opening the shutter or bringing the heating source nearer to the surface.

e) Measure the temperature history in the temperature-rising process and confirm that the specified heating rate is achieved.

f) Set the temperature within a ±5 % range of the maximum heating temperature to be set and maintain this temperature for the specified period of time.

g) Measure the temperature of the test piece or heat flux meter and confirm that the temperature gradient has been achieved.

h) Stop the heating of the heating surface of the test piece by closing the shutter or moving the heating source away from the surface.

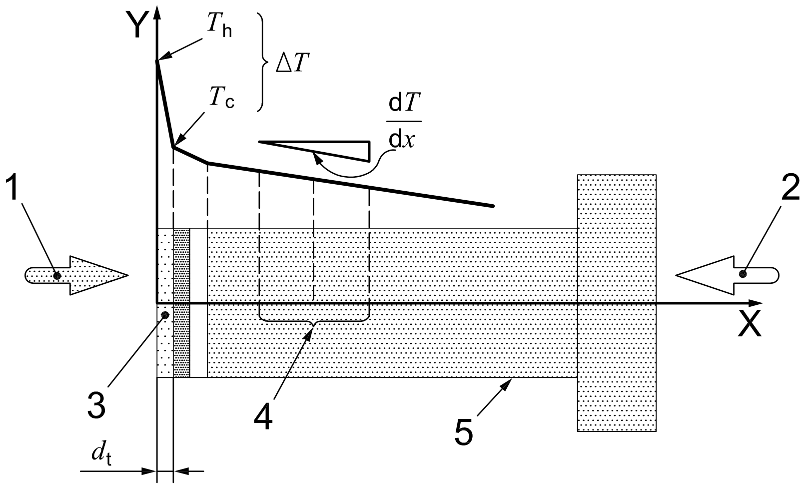
i) If necessary, allow the heating surface to cool.

j) Measure the temperature history as the temperature drops and confirm that the cooling rate is as specified.

k) Repeat steps d) to j) as specified.

6.3 Calculation

The temperature distribution for a representative temperature gradient is shown in Figure 4.



Key

1 heating
2 cooling
3 test piece
4 thermocouple
5 heat flux meter
X distance, x (mm)
Y temperature, T (°C)
d_t top coat thickness (mm)
$\frac{dT}{dx}$ average temperature gradient (°C/mm) in the heat flux meter as measured with the thermocouple
T_h top coat surface temperature (°C)
T_c top coat backside temperature (°C)

Figure 4 — Schematic diagram of temperature distribution under temperature-gradient condition

The heat flux and equivalent effective thermal conductivity shall be calculated as follows.

Note that the numerical values shall be rounded to two decimal places in accordance with ISO 80000-1.

a) Temperature difference of the top coat

The temperature difference of the top coat is calculated using Equation (1):

$$\Delta T = T_h - T_c \qquad (1)$$

where

ΔT is the temperature difference of the top coat, in degrees Celsius;

T_h is the top coat surface temperature, in degrees Celsius;

T_c is the top coat backside temperature, in degrees Celsius.

NOTE 1 This may be substituted by the values extrapolated from the readings of the heat-flux-meter thermocouples or the measured value of the substrate temperature displayed by the thermocouple (see Annex A).

b) Heat flux

When the heat flux meter is used, heat flux is calculated from Equation (2):

$$q = -\lambda_s \times \frac{dT}{dx} \tag{2}$$

where

q is the heat flux, in watts per square millimetre;

λ_s is the thermal conductivity, in watts per millimetre kelvin, of the heat flux meter;

$\frac{dT}{dx}$ is the average temperature gradient, in degrees Celsius per millimetre, in the heat flux meter, as measured with the thermocouple.

NOTE 2 For the thermal conductivity of the heat flux meter, use the thermal conductivity corresponding to the average of all the temperatures measured with the heat flux meter.

c) Equivalent effective thermal conductivity of top coat

When the heat flux meter is used, the equivalent effective thermal conductivity is calculated from Equation (3):

$$\lambda_{eff} = \frac{q \times d_t}{T_h - T_c} \tag{3}$$

where

λ_{eff} is the equivalent effective thermal conductivity of the top coat, in watts per millimetre per degree Celsius;

q is the heat flux, in watts per square millimetre;

d_t is the top coat thickness, in millimetres;

T_h is the top coat surface temperature, in degrees Celsius;

T_c is the top coat backside temperature, in degrees Celsius.

6.4 Evaluation

6.4.1 General

Evaluation of the thermal-barrier performance and cyclic heat resistance shall be as follows. Note that, in both cases, a test piece with damage occurring from the test-piece edge shall not be used for evaluation.

6.4.2 Evaluating the thermal-barrier performance

The thermal-barrier performance of the test piece shall be evaluated in terms of the maximum surface temperature, the temperature difference between heating and cooling surfaces, and the equivalent effective thermal conductivity. The damage-occurrence time point shall be assumed to be a point at which the equivalent effective thermal conductivity indicates a sudden decrease, or the number of measured acoustic emissions increases suddenly.

6.4.3 Cyclic heat resistance

The cyclic heat resistance of the test piece shall be evaluated in terms of the top coat surface maximum temperature, the maximum temperature difference between the top coat surface and backside, and the number of heating cycles needed to reach the damage tolerance limit. The damage tolerance limit shall be established based on agreement between the parties involved, but is normally determined to be the point at which the spalling area ratio has reached 20 %, as assessed by visual observation.

When spalling has not occurred, it is determined that no damage occurs at this number of heating cycles.

Note that the moment when spalling occurs may be considered the time when the equivalent effective thermal conductivity decreases suddenly or the number of measured acoustic emissions increases suddenly.

7 Test report

When a test report is necessary, the report items shall be agreed upon between the parties and selected from the items listed below.

a) Test piece

   1) Substrate material

   2) Coating material

   3) Coating method

   4) Shape and dimensions of test piece

   5) Coating thickness

   6) Number of test pieces

   7) Bonding material

   8) Bonding thickness

b) Test equipment

   1) Heating unit

   2) Cooling unit

   3) Test block

      i) Heating flux meter (material, shape, dimensions, including the diameter and position of thermocouple holes)[1]

      ii) Test environment

c) Testing conditions (see Figure 5)

   1) Heating-unit working conditions

   2) Cooling-unit working conditions

1) Items shall be reported in the case of data acquisition.

3) Heating time

4) Cooling time

5) Top coat maximum temperature

6) Top coat minimum temperature

7) Top coat maximum temperature holding time

8) Top coat minimum temperature holding time

9) Initial heating rate (= initial temperature increment/arrival time)

10) Average heating rate (= temperature increment during heat cycle/average arrival time)

11) Average cooling rate (= temperature decrement during heat cycle/average arrival time)

12) Temperature increase time

13) Temperature decrease time

14) Initial temperature increase time

d) Test result

1) Heat flux

2) Equivalent effective thermal conductivity of top coat[1)]

3) Temperature difference in top coat

4) Number of heating cycles needed to reach the damage tolerance limit of top coat

5) Top coat damage or damage pattern (cracking, spalling, dislodgment)

e) Date of test

f) Additional statement

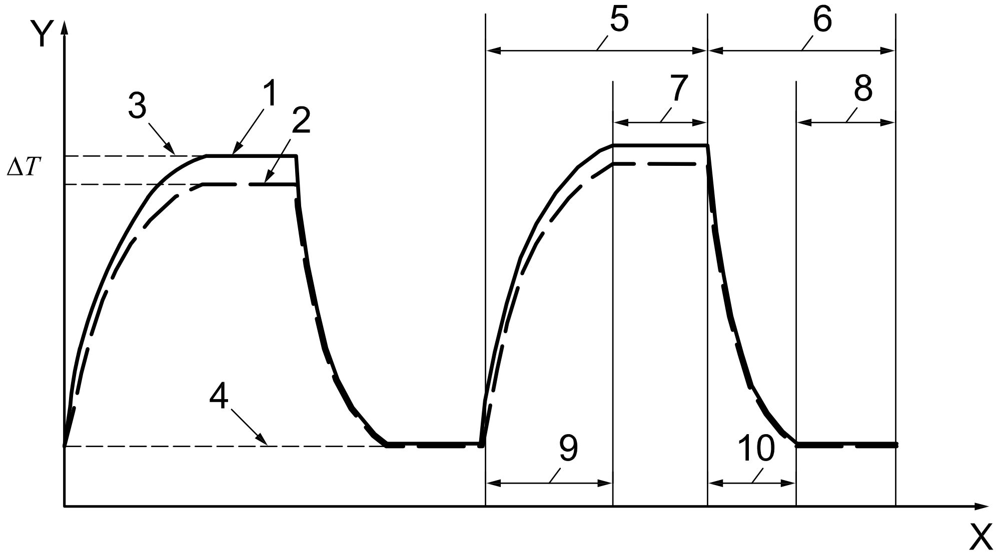
The following additional statement should be included in the test report.

1) Temperature-history curve

2) Change of the equivalent effective thermal conductivity according to the number of cycles[1)]

3) Photograph or illustration showing typical damage pattern of test piece

4) Typical results of acoustic emission measurement results[1)]



Key

1 top-surface temperature of TBC
2 backside temperature of TBC
3 maximum temperature
4 minimum temperature
5 heating time
6 cooling time
7 maximum temperature holding time
8 minimum temperature holding time
9 temperature increase time
10 temperature decrease time
X time, t (s)
Y temperature, T (°C)

Figure 5 — Temperature history of top coat during cyclic heating test

Annex A
(informative)

Calculation of temperature at the bottom side of the top coat

T_c can be calculated from the following simultaneous equations:

$$T_3 = T_4 - \frac{dT}{dx} \cdot d_4$$

$$q = \lambda_{eff} \cdot \frac{T_h - T_c}{d_t} = \lambda_{flux} \cdot \left(-\frac{dT}{dx}\right) = \lambda_{braz} \cdot \frac{T_2 - T_3}{d_{braz}} = \lambda_{sub} \cdot \frac{T_1 - T_2}{d_{sub}} = \lambda_b \cdot \frac{T_c - T_1}{d_b}$$

Then, T_c is expressed as follows:

$$T_c = T_4 - \frac{dT}{dx} \cdot d_4 + \lambda_{flux} \cdot \left(-\frac{dT}{dx}\right) \cdot \left(\frac{d_{braz}}{\lambda_{braz}} + \frac{d_{sub}}{\lambda_{sub}} + \frac{d_b}{\lambda_b}\right)$$

where

q is the heat flux, in watts per square millimetre;

T_h is the temperature of the top side of the top coat (measured), in degrees Celsius;

T_c is the temperature of the bottom side of the top coat, in degrees Celsius;

T_1 is the temperature of the interface between the bond coat and the substrate, in degrees Celsius;

T_2 is the temperature of the interface between the substrate and the brazed layer, in degrees Celsius;

T_3 is the temperature of the interface between the brazed layer and the heat flux meter, in degrees Celsius;

T_4 is the temperature measured by the thermocouple, in degrees Celsius;

d_t is the thickness of the top coat, in millimetres;

d_b is the thickness of the bond coat, in millimetres;

d_{sub} is the thickness of the substrate, in millimetres;

d_{braz} is the thickness of the bonding layer, in millimetres;

d_4 is the distance from the top surface of the heat flux meter to the thermocouple, in millimetres;

dT/dx is the average temperature gradient along the central axis of the heat flux meter, in degrees Celsius per millimetre (known);

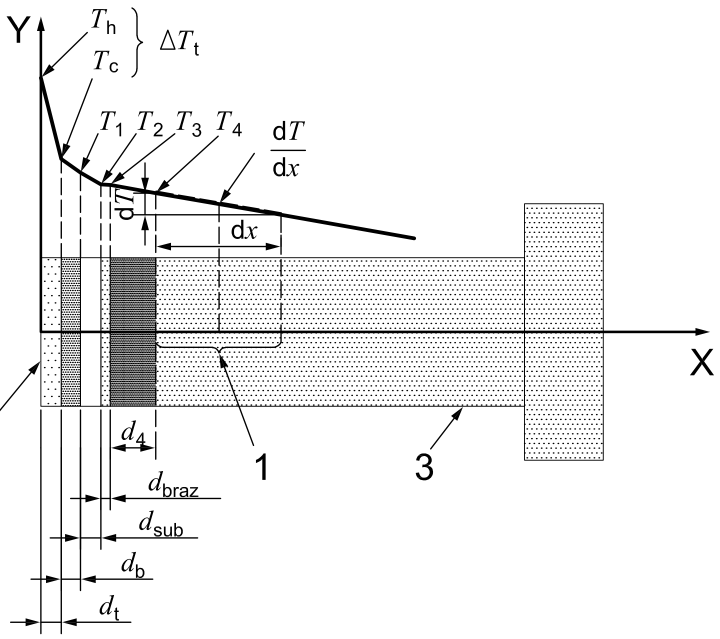
λ_{eff} is the equivalent effective thermal conductivity of the top coat, in watts per millimetre per degree Celsius;

λ_b is the thermal conductivity of the bond coat, in watts per millimetre per degree Celsius;

λ_{sub} is the thermal conductivity of the substrate, in watts per millimetre per degree Celsius;

λ_{braz} is the thermal conductivity of the brazing layer, in watts per millimetre per degree Celsius;

λ_{flux} is the thermal conductivity of the heat flux meter, in watts per millimetre per degree Celsius.



Key

1 thermocouple
2 test piece
3 heat flux meter
X distance, x (mm)
Y temperature, T (°C)

Figure A.1 — Calculation procedure of temperature at the bottom side of the top coat

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