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Metallic and other inorganic coatings — Test methods for measuring thermal cycle resistance and thermal shock resistance for thermal barrier coatings

Revêtements métalliques et autres revêtements inorganiques — Méthodes d'essai pour mesurer la résistance au cyclage thermique et la résistance au choc thermique des revêtements barrières thermiques

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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.

ISO 14188 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

Introduction

Thermal barrier coatings are highly advanced material systems, generally applied to surfaces of hot-section parts made of nickel or cobalt based superalloys, such as blades, vanes and combustors in gas turbines and aero-engines, operated at elevated temperatures.

The purpose of these coatings is to insulate metallic components for an extended period at elevated temperatures by employing thermally insulating materials which can sustain an appreciable temperature difference between load bearing alloys and coating surfaces. By shielding these parts, these coatings permit the use of high operating temperatures by restricting exposure of structural parts to these temperatures, thereby extending their lives.

This International Standard specifies test methods, applicable to these thermal barrier coatings, for measuring thermal cycle resistance and thermal shock resistance using appropriate heating and cooling procedures.

Metallic and other inorganic coatings — Test methods for measuring thermal cycle resistance and thermal shock resistance for thermal barrier coatings

1 Scope

This International Standard specifies test methods applicable to thermal barrier coatings for measuring thermal cycle resistance by using steady cyclical heating and cooling procedures, and for measuring thermal shock resistance using a heating and quenching technique.

These measurements are used for the evaluation of durability of thermal barrier coatings to thermal strain.

This International Standard is applicable for screening thermal barrier coating systems including materials and processing, and not for controlling the thermal spraying processes.

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 14232, *Thermal spraying — Powders — Composition and technical supply conditions*

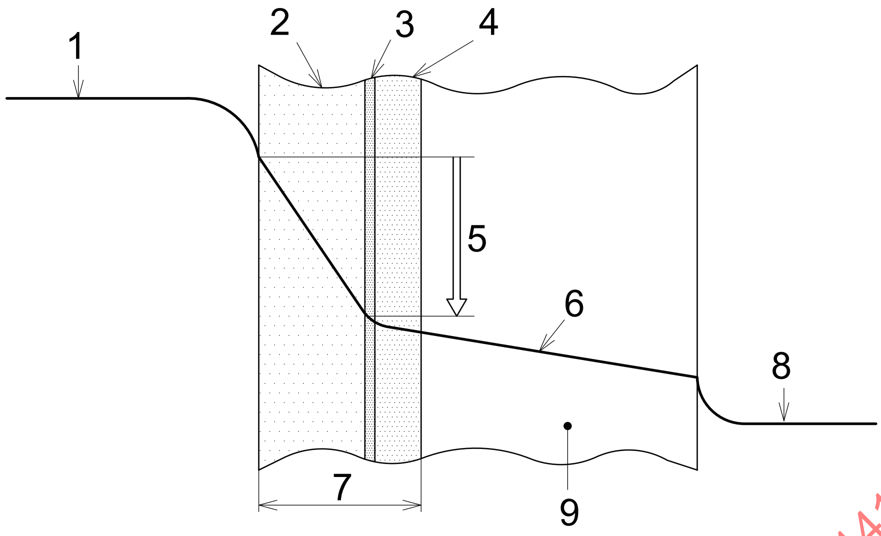
ISO 14916, *Thermal spraying — Determination of tensile adhesive strength*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
thermal barrier coating
TBC
two-layer coating consisting of a metallic bond coat and an oxide top coat, in order to reduce heat transfer from outside the top coat through the coating to the base material

NOTE When a TBC is exposed to operating at a high temperature, thermally grown oxide (TGO) develops on the top of the bond coat. The substrate is a typically heat-resistant high nickel based alloy class, UNS N 06002 type of material. The coatings, illustrated in Figure 1, are applied by using physical or chemical vapour deposition or by thermal spray processes, such as plasma spray and high velocity oxy-fuel (HVOF) spray conforming to ISO 14232.



Key

1 combustion gas temperature
2 top coat
3 TGO
4 bond coat
5 thermal barrier effect
6 temperature
7 TBC
8 cooling air temperature
9 substrate

Figure 1 — Diagrammatic view of the section and effects of TBC

3.2
thermally grown oxide
TGO
oxide grown between top and bond coat when the coating system is heated

3.3
ratio of spalling areas
proportion of the total spalling area relative to the effective area of the TBC

3.4
critical number of cycles to spalling
number of thermal cycles immediately prior to achieving 30 % in ratio of spalling area

NOTE In the assessment of spalling area, areas of delamination or cracking are excluded.

3.5
thermal shock temperature difference
difference in temperatures between heating and cooling water treatments of the test pieces in thermal shock tests

3.6
thermal shock resistance
thermal shock temperature difference that causes a reduction of tensile adhesive strength immediately prior to a decrease of 30 % relative to the tensile adhesive strength in the absence of thermal shock

4 Principle

The test methods include the thermal cycle resistance test by using steady cyclical heating and cooling procedures and consist of measuring ratio of spalling areas, and the thermal shock resistance test by measuring tensile adhesive strength following application of thermal shock. Thermal shock is applied by heating the test pieces in a furnace to the appropriate temperature and quenching in water. The coatings are applied by physical, chemical vapour deposition or by thermal spray processes such as plasma spray and high velocity oxy-fuel (HVOF) spray by using feedstock powders (ISO 14232).

5 Test methods

5.1 General

a) All measurements related to physical quantities, dimensions and quantities and unit systems shall be carried out in accordance with appropriate International Standards (see Bibliography).

b) When ordering testing in accordance with this International Standard, agreement between the contracting parties are needed in writing in contract or purchase order, concerning the following:

   1) handling and sectioning procedures, method of application of coatings, whether the coating surface is to be used as coated;

   2) for the thermal cycle test, selection of test pieces, position and size of piercing on test pieces, number of and any interruption of thermal cycles, and upper limit of high temperature, heating time, holding time at high temperature, cooling time and holding time at low temperature of a thermal cycle.

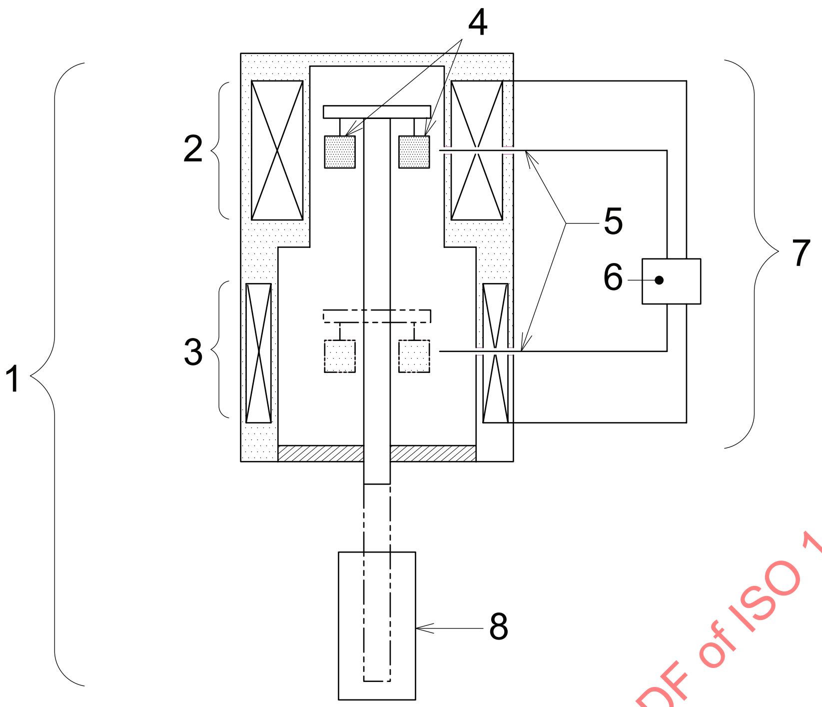
5.2 Thermal cycle resistance method

5.2.1 General

The test method covers a thermal cycle resistance test by using steady cyclical heating and cooling procedures and consists of measuring ratio of spalling areas. This method is useful in the selection of materials, coating processes and process conditions in the TBC system used in gas turbines operating at elevated temperatures.

5.2.2 Apparatus

The apparatus consists of a heating and cooling device and temperature control system, and is shown in Figure 2.

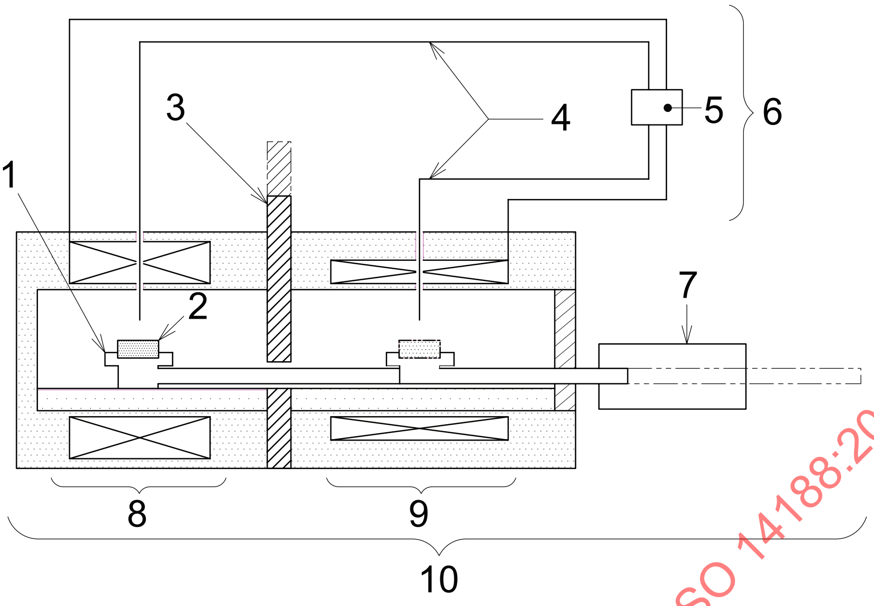


a) Vertical type

Key

1 heating and cooling device
2 high-temperature bath
3 low-temperature bath
4 test piece
5 thermocouple
6 temperature controller
7 temperature control system
8 test-piece-moving device

Figure 2 — Typical device for thermal cycle resistance method (*continued*)



b) Horizontal type

Key

1 test piece support
2 test piece
3 shield plate (up/down possible)
4 thermocouple
5 temperature controller
6 temperature control system
7 test-piece-moving device
8 high-temperature bath
9 low-temperature bath
10 heating and cooling device

Figure 2 — Typical device for thermal cycle resistance method

5.2.2.1 Heating and cooling device

a) The heating and cooling device consists of high- and low-temperature baths respectively. The atmosphere in both baths is air.

b) The test piece is placed alternately in the two baths which are fixed; or alternatively, the test piece is fixed and the baths are alternated.

c) The test pieces may be directly exposed to ambient air instead of using the low-temperature bath.

d) The temperature control system consists of the temperature controller and thermocouples (see Figure 2). The temperature is regulated to ensure that the temperature of the test pieces is within the range given in Table 1.

Table 1 — Permissible temperature deviation from the target test temperature of test pieces

Test temperature °C	Permissible deviation °C
Room temperature to ≤ 600	±3
> 600 to ≤ 800	±4
> 800 to ≤ 1 000	±5
> 1 000	±7

5.2.3 Test pieces

5.2.3.1 Test pieces

The test pieces shall be of the flat plate type (Figure 3) or the disk type (Figure 4). To facilitate handling, the test pieces may be pierced.

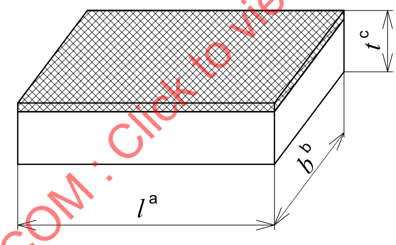
Chamfering or rounding of the substrate edges shall appropriately be applied in order to avoid stress concentration at these locations.

5.2.3.2 Measurement of dimensions

Measure the dimensions of the parts to the nearest 0,1 mm using appropriate instruments [see 5.1.a)].

5.2.3.3 Measurement of mass of test pieces

Weigh the test pieces to the nearest 1 mg using appropriate instruments [see 5.1.a)].



Key

l length

b width

t total thickness

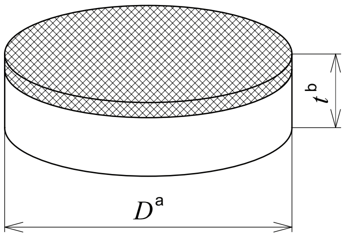
[a] 20 mm to 100 mm

[b] 20 mm to 50 mm

[c] 1,5 mm to 12,7 mm

The shaded area is covered with the TBC.

Figure 3 — Shape and dimensions of flat-plate-type test piece



Key

D outside diameter

t total thickness

[a] 20 mm to 40 mm

[b] 1,5 mm to 5,0 mm

The shaded area is covered with the TBC.

Figure 4 — Shape and dimensions of disk-type test piece

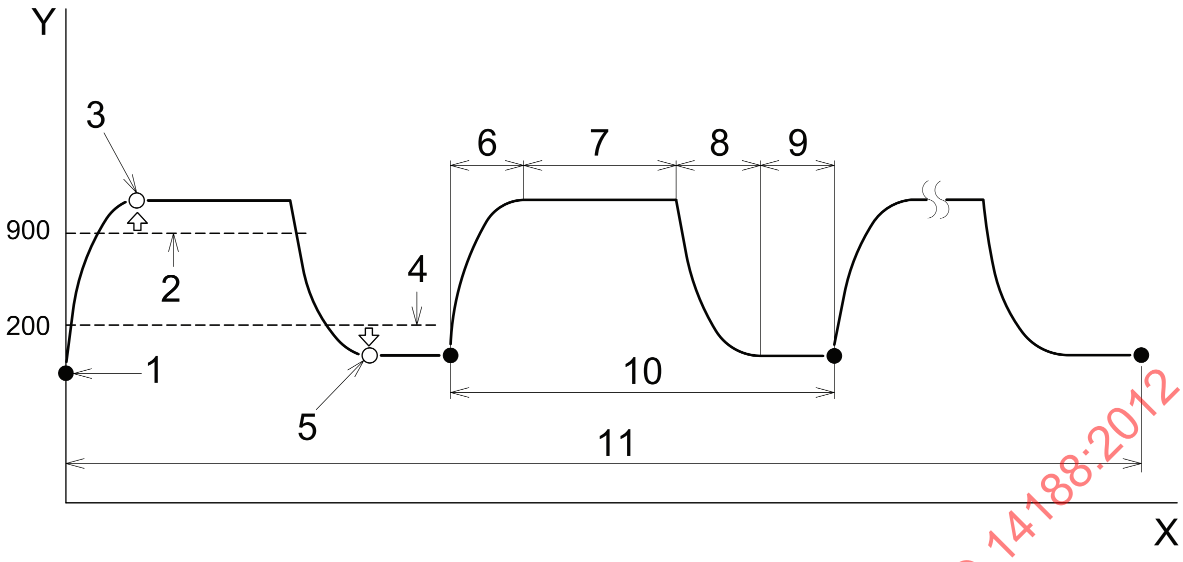
5.2.4 Test procedures

5.2.4.1 Heating and cooling methods

Test pieces are held in the low-temperature bath before transferring them to the high-temperature bath and are held there for a specified duration. Once the test pieces have achieved stability, they are then relocated to the low-temperature bath and held there for a specified duration. A thermal cycle, shown in Figure 5, therefore, consists of a period of stability in the low-temperature bath and subsequently a period of stability in the high-temperature bath at the specified temperature.

The temperatures at the high and low temperature are calibrated by attaching thermocouples to dummy test pieces for adjustment to the specified temperature prior to the actual test runs.

The number of thermal cycles is referred to in 5.1 b) 2).



Key

1 start
2 lower limit
3 temperature at high temperature
4 upper limit
5 temperature at low temperature
6 heating time
7 holding time at high temperature
8 cooling time
9 holding time at low temperature
10 1 cycle
11 *N* cycles (*N*: 1, 2, 3, …)
X time
Y temperature, *T* (°C)

Figure 5 — A typical thermal cycle

5.2.4.2 Heating and cooling conditions

a) Table 1 shows the permissible temperature deviation from the target test temperature. For the high test temperature, the lower limit shall be 900 °C; and for the low test temperature, the upper limit shall be 200 °C. For the upper limit of the high-temperature range, see 5.1.b) 2).

b) An example of a temperature pattern for a thermal cycle, shown in Figure 5, consists of 10 min for a temperature rise from 200 °C to 900 °C, 30 min for maintaining the test piece at the high temperature, 15 min for the temperature to drop from 900 °C to 200 °C, and 5 min for maintaining the test piece at the low temperature. Time conditions in a thermal cycle may be varied [see 5.1 b) 2)].

c) Multiple test pieces may be treated simultaneously to enhance the efficiency of the test procedures.

5.2.4.3 Examination for spalling during thermal cycle resistance test

a) The test may be interrupted after completion of an agreed number of cycles and the test piece is allowed to cool down to 200 °C and examined for any spalling. Once the number of cycles up to the stage when spalling occurs has been calculated or estimated, tests may be continued until close to the completion of the expected number of cycles.

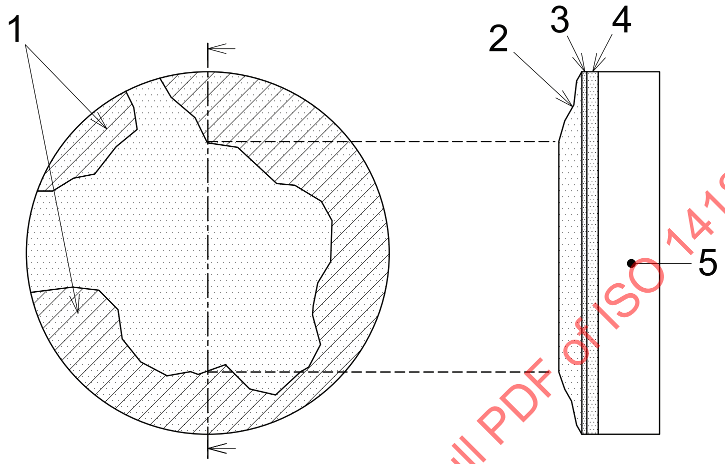
b) Inspect the test piece for cracks in the TBC or spalling visually with normal or corrected vision.

c) If cracks or spalling are detected, record them photographically.

5.2.4.4 Measurement of ratio of spalling areas

a) Assess the spalling area in the TBC using, for example, image analysis from the record [see 5.2.4.3 c)]. For multiple spalling sites, the spalling area shall be the total of all spalling locations. Figure 6 illustrates typical spalling sites and total spalling area.

b) For the occurrence of severe oxidation of the test pieces, weigh them and assess the mass loss (see 5.2.3.3).



Key

1 spalling area

2 top coat

3 TGO

4 bond coat

5 substrate

The view of the test piece surface is shown on the right and the sectional view of the middle portion is shown on the left.

Figure 6 — Typical spalling sites and cross-sectional view of middle part

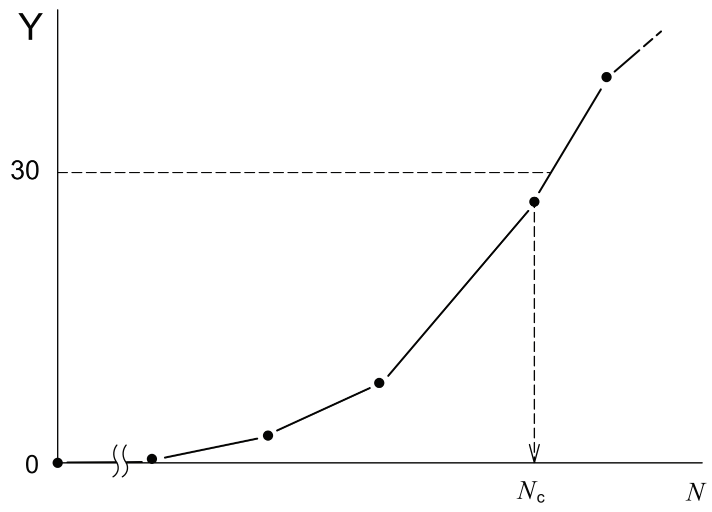
5.2.4.5 Determination of critical number of cycles to spalling

The critical number of cycles to spalling is the number of thermal cycles immediately prior to achieving an increase in the ratio of spalling area by 30 %.

Use three or more test pieces for the same test conditions to determine the critical number of cycles to spalling. The test is to be considered as completed when the ratio of spalling areas exceeds 30 % of the TBC or the number of thermal cycles reaches the number specified in 5.1 b) 2).

If the TBC has spalled more than 30 % in area during one thermal cycle, the critical number of cycles is to be considered as one.

The relationship between the number of thermal cycles and the ratio of spalling areas (see 3.3) is shown in Figure 7.



Key

N_c critical number of cycles to spalling

N number of thermal cycles (time)

Y ratio of spalling area (%)

Figure 7 — An example of the relationship between the number of thermal cycles and ratio of spalling areas

5.2.4.6 Examination of the test piece after completion of the thermal cycle resistance test

a) Examine the test pieces in accordance with 5.2.4.3.

b) Examine the microstructure of the cross-section of the test pieces at an appropriate magnification and record it photographically.

5.3 Thermal shock resistance method

5.3.1 General

Thermal shock is used to evaluate the effectiveness of resistance to spalling of the TBC and to measure the tensile adhesive strength of test pieces under special environmental conditions, such as emergency shutdown in gas turbines, etc.

5.3.2 Apparatus

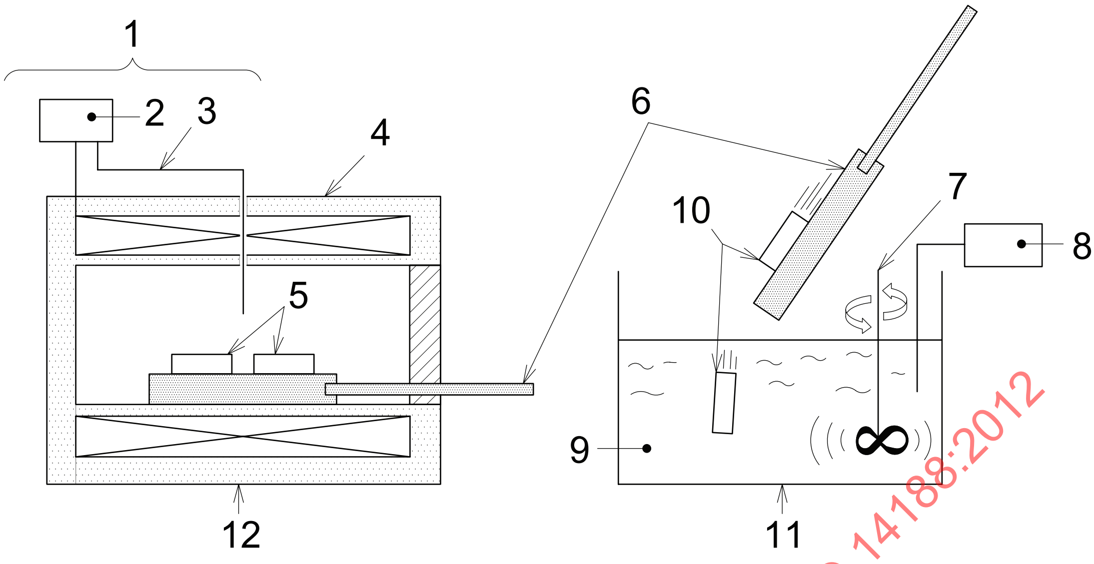
The apparatus consists of a heating device, temperature control system and a cooling water bath, and is shown in Figure 8.

5.3.2.1 Heating device and cooling water bath

a) The heating device consists of the temperature control system (see 5.2.2.1 d)] with a furnace capable of heating in the temperature range shown in Table 1 and having a test chamber allowing a flow of ambient air.

   A muffle electric resistance furnace may also be used.

b) The cooling water bath consists of a water bath with a thermometer to measure the water temperature and a stirrer, and is of an adequate capacity to cool the entire test piece rapidly.



Key

1 temperature control system
2 temperature controller
3 thermocouple
4 heating furnace
5 test piece
6 test piece support
7 stirrer
8 thermometer
9 water
10 test piece
11 cooling water bath
12 heating device

Figure 8 — Typical device for thermal shock resistance test

5.3.3 Test pieces

a) The test pieces shall be of the disk type (Figure 9). The substrates are to be coated with the TBC on both sides. If application of coating on both sides is not possible, only one side may be coated.

Chamfering or rounding of the substrate edges shall be applied appropriately in order to avoid stress concentration at these locations.

b) For all other conditions, see 5.1.