
**Metallic and other inorganic
coatings — Measurement of the linear
thermal expansion coefficient of
thermal barrier coatings**

*Revêtements métalliques et autres revêtements inorganiques —
Mesure du coefficient de dilatation thermique linéaire des
revêtements barrières thermiques*

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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.

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).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

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

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

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

The function of these coatings is to protect metallic components for extended periods at elevated temperatures by employing thermally insulating materials that can sustain an appreciable temperature difference between load bearing alloys and coating surfaces. These coatings permit the high-temperature operation by shielding these components, thereby extending their lives.

Although the linear thermal expansion coefficient is an important property of thermal barrier coatings, the existing International Standard (e.g. ISO 17562) describes only a method for measuring this parameter for monolithic ceramics.

This document specifies a method for measuring the linear thermal expansion coefficient of the ceramic top coat for thermal barrier coating.

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Metallic and other inorganic coatings — Measurement of the linear thermal expansion coefficient of thermal barrier coatings

1 Scope

This document specifies a method for measuring the reproducible linear thermal expansion coefficient of ceramic top coats (TCs) for thermal barrier coatings (TBCs) up to 1 300 °C.

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.

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 17139, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Thermophysical properties of ceramic composites — Determination of thermal expansion*

ISO 17562, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for linear thermal expansion of monolithic ceramics by push-rod technique*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 17139 and the following 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/>

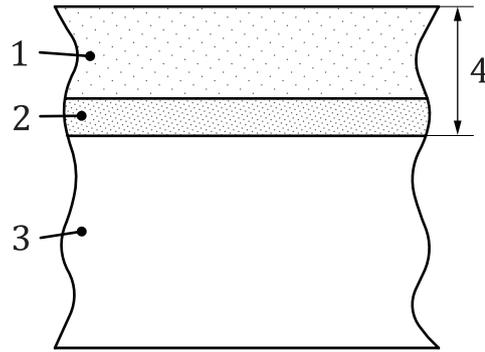
3.1

thermal barrier coating

TBC

two-layer coating consisting of a metallic bond coat (BC) and a ceramic top coat (TC), in order to reduce heat transfer from outside the top coat through the coating to the substrate

Note 1 to entry: See [Figure 1](#).



Key

- | | | | |
|---|----------------|---|-------------------------------|
| 1 | top coat (TC) | 3 | substrate |
| 2 | bond coat (BC) | 4 | thermal barrier coating (TBC) |

Figure 1 — Diagrammatic view of a section of a TBC

[SOURCE: ISO 14188:2012, 3.1, modified — “ceramic top coat” has replaced “oxide top coat”, “substrate” has replaced “base material”, and a different note to entry and figure has been used.]

3.2 mean linear thermal expansion coefficient

$\alpha(T)$

linear thermal expansion between temperatures T_1 and T_2 divided by the temperature increment from T_1 to T_2 and the length at room temperature T_0 , as shown by the formula:

$$\alpha(T) = \frac{L(T_2) - L(T_1)}{L(T_0)} \times \frac{1}{(T_2 - T_1)}$$

where

$$T = \frac{T_2 + T_1}{2};$$

$L(T_0)$ is the initial length of the specimen at room temperature;

$L(T_1)$ is the length of the specimen at temperature T_1 ;

$L(T_2)$ is the length of the specimen at temperature T_2 .

4 Principle

Ceramic TCs exhibit an unstable linear thermal expansion coefficient during the initial few heating cycles, as shown in [Annex A](#). Therefore, the measurement shall be repeated until the reproducible linear thermal expansion coefficient is obtained. The fundamental procedures are shown in [Figure 2](#). A specimen of a TC, prepared as described in [Clause 6](#), that constitutes a TBC is heated to a specified temperature at a controlled heating rate. During the heating, the linear thermal expansion coefficients of the specimen are measured based on the change of the length and the temperature of the specimen. The measurement shall be repeated until the criterion to finish the measurement mentioned in [Clause 7](#) is satisfied.

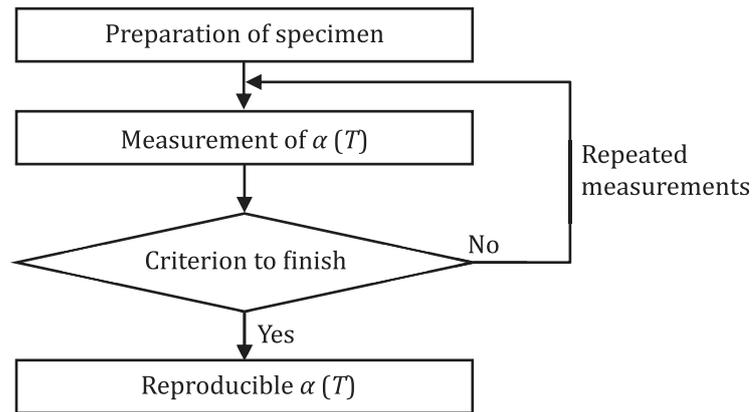


Figure 2 — Fundamental procedures for determining the linear thermal expansion coefficient

5 Apparatus for measuring the linear thermal expansion coefficient

5.1 Apparatus

An example of the apparatus for measuring the linear thermal expansion coefficient is schematically shown in Figure 3. The apparatus shall be specified in accordance with ISO 17139 and shall be calibrated using the reference specimen (see 6.2). Care shall be taken to ensure that the thermocouple tip is in close proximity to the specimen in accordance with ISO 17562.

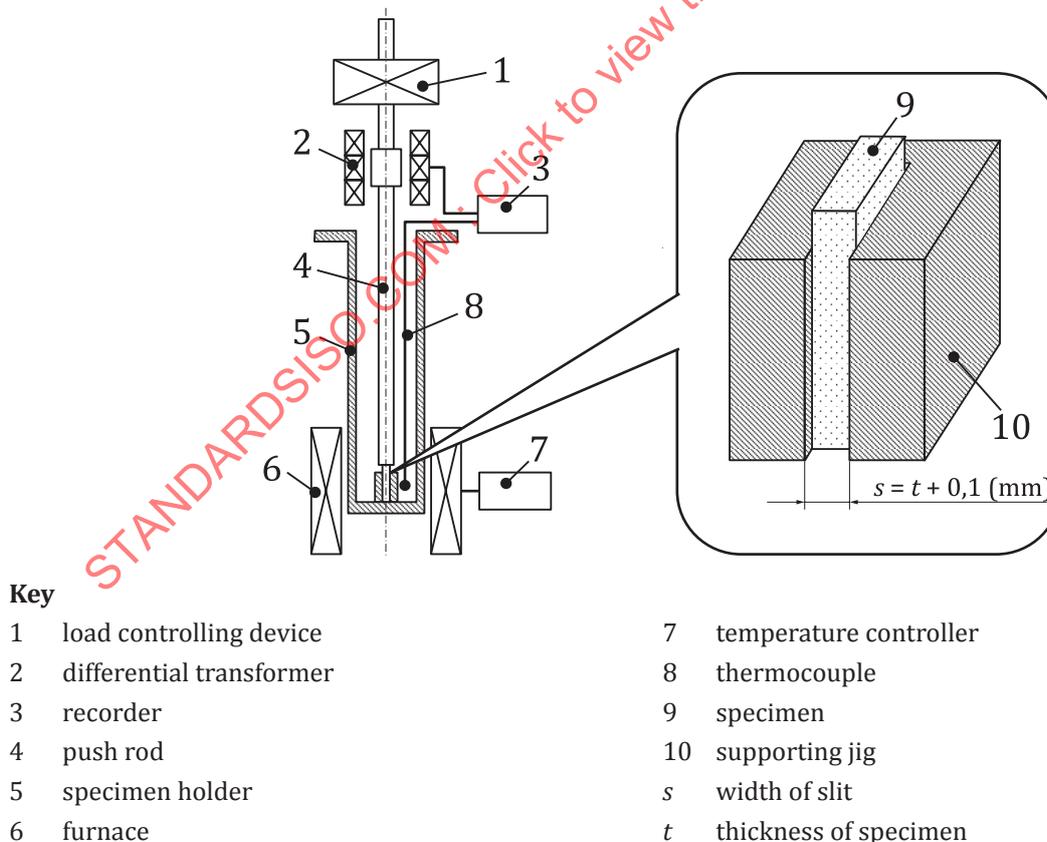


Figure 3 — Typical apparatus for measuring the linear thermal expansion coefficient

5.2 Supporting jig

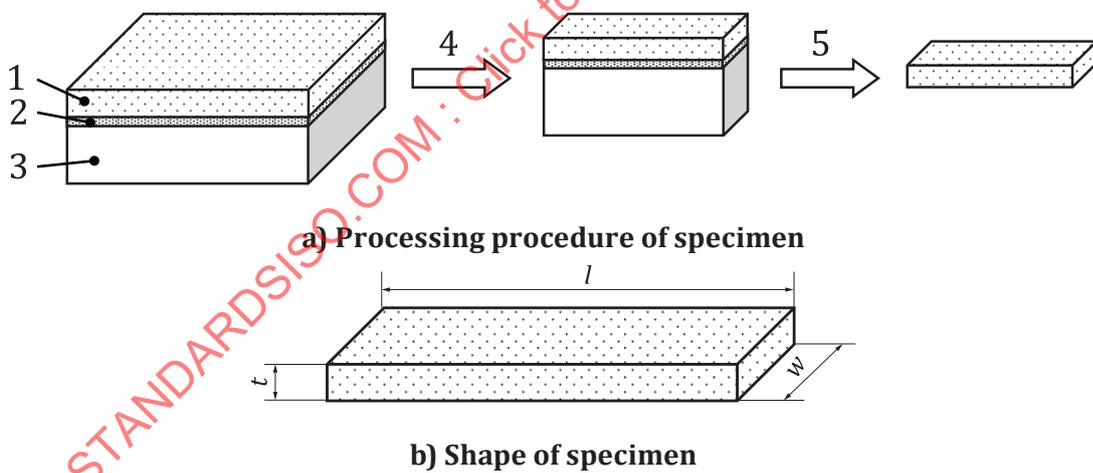
The supporting jig is specified as follows.

- a) The supporting jig should be used in order to prevent the specimen from turning over or tilting, but it shall allow free dilatation of the specimen.
- b) The material of the supporting jig shall not react with the specimen and the specimen holder, and shall be of the same material as that of the specimen holder. High purity alumina is recommended for measurements.
- c) The shape and dimensions of the supporting jig depend on the apparatus used.
- d) The width of slit for inserting the specimen shall be larger than the thickness of the specimen by about 0,1 mm.

6 Specimen

6.1 Test specimen

- a) From a TBC formed on a substrate, a section shall be cut out by machining, and the TC specimen in plane which is free standing shall be chemically stripped or mechanically machined from the substrate, as shown in [Figure 4](#).
- b) The specimen shall be a plate 10 mm or more in length, 5 mm or more in width, and 0,5 mm or more in thickness.
- c) The thickness shall be decided so that the specimen does not warp.



Key

- | | | | |
|---|-----------------------------|-----|-----------|
| 1 | top coat (TC) | l | length |
| 2 | bond coat (BC) | w | width |
| 3 | substrate | t | thickness |
| 4 | machining | | |
| 5 | removal of BC and substrate | | |

Figure 4 — Processing procedure, shape and dimensions of specimen

6.2 Reference specimen

The reference specimen shall be specified in accordance with ISO 17139.

7 Measuring procedure

The measuring procedure shall be as follows.

- a) The length, width and thickness of the specimen shall be measured in accordance with ISO 3611. The length shall be measured with the uncertainty of less than 0,1 % using a micrometer.
- b) The supporting jig shall be placed in the specimen holder.
- c) The specimen shall be inserted into the supporting jig. The contact force of the push rod to the specimen shall be adjustable between 0,1 N to 1 N.
- d) The specimen shall be heated at a constant rate between 1 °C/min and 5 °C/min.
- e) The temperature and the change of length of the specimen shall be recorded as finely as possible in order to increase the accuracy of calculation of the linear thermal expansion coefficient.
- f) The temperature increment for $\alpha(T)$ shall be approximately 50 °C.
- g) The measurement shall be repeated without dismounting the specimen from the apparatus until the criterion to finish the measurement is satisfied.
- h) The criterion to finish the measurement is expressed by [Formula \(1\)](#). Assuming that the difference between $\alpha(T)_N$ and $\alpha(T)_{N-1}$ relative to the average value of $\alpha(T)_N$ and $\alpha(T)_{N-1}$ should be within 5 % as the criterion, the criterion value Z_0 is 5 %.

$$Z(T) = \frac{|\alpha(T)_N - \alpha(T)_{N-1}|}{\{\alpha(T)_N + \alpha(T)_{N-1}\}/2} \times 100 \leq Z_0 \quad (1)$$

where

$Z(T)$ is the value of the parameter (in %);

Z_0 is the criterion value (in %);

$\alpha(T)_N$ is the mean linear thermal expansion coefficient in the N 'th measurement (in °C⁻¹);

$\alpha(T)_{N-1}$ is the mean linear thermal expansion coefficient in the $(N-1)$ 'th measurement (in °C⁻¹).

8 Test report

The test report shall contain the following items:

- a) the International Standard used;
- b) specimen:
 - 1) material and coating conditions;
 - 2) shape and dimensions;
- c) measurement conditions:
 - 1) type of measuring apparatus;
 - 2) materials of supporting jig, specimen holder and push rod;

- 3) applied force, heating rate, maximum temperature and atmosphere;
 - 4) range of parameters $Z(T)$ and Z_0 ;
- d) results:
- 1) number of measurements;
 - 2) linear thermal expansion curve;
 - 3) linear thermal expansion coefficient;
 - 4) deviations from the procedure and unusual features observed if any;
 - 5) the date of the test.

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