

Testing of engineering ceramics —

Part 4: Thermo-physical properties —

Section 4.1 Method for determination of thermal expansion

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- British Metallurgical Plant Constructors' Association
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- Electricity Supply Industry in England and Wales
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Contents

	Page
Committees responsible	Inside front cover
Foreword	ii
1 Scope	1
2 Definition	1
3 Principle	1
4 Apparatus	1
5 Test pieces	2
6 Procedure	2
7 Calculation of results	3
8 Reporting of results	3
9 Test report	3
Appendix A Calibration of apparatus (direct-measuring instruments)	5
Appendix B Calibration of apparatus (differential type)	6
Appendix C Thermal expansion reference data	6
Figure 1 — Schematic diagrams of apparatus arrangements	8
Figure 2 — Suitable constructions of test piece holders in silica and alumina	9
Figure 3 — Silica ball test piece support and carrier	10
Figure 4 — Arrangement of differential thermocouples to monitor temperature distribution in the test piece	11
Table 1 — Thermal expansion reference data	7
Publications referred to	Inside back cover

Foreword

This Section of BS 7134 has been prepared under the direction of the Refractory Products Standards Policy Committee. It is one of a series of Sections describing the testing of ceramics used in the engineering industry.

This Section is to be read in conjunction with BS 7134-0 “*Introduction and guide to sampling*” which describes the type of products which may be tested and also lists the Parts and Sections of BS 7134.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Section of BS 7134 describes the method for the determination of the linear thermal expansion characteristics of engineering ceramics up to a maximum temperature of 1 400 °C (see 4.2) and to an accuracy of not less than $\pm 0.1 \times 10^{-6} \text{ K}^{-1}$ over a temperature interval of 100 K.

The method describes general principles of construction, calibration and operation of suitable apparatus. Specific details, including test piece dimensions, depend on the design of the apparatus. Methods of calibration are given in Appendix A and Appendix B. Thermal expansion reference data are given in Appendix C.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definition

For the purposes of this Section of BS 7134, the following definition applies.

thermal expansion

the proportional extension which occurs when a material is heated

3 Principle

A test piece is heated and subsequently cooled, either at a specified uniform rate or using defined temperature increments. Its change of length and its temperature are measured continuously during the heating and cooling. The percentage expansion or contraction over the required temperature range is calculated and the results presented both as a mean linear thermal expansion coefficient over a chosen temperature range, and as a graph.

4 Apparatus

4.1 General. The apparatus shall conform to the specification given below and be capable of calibration according to the procedures given either in Appendix A for direct measurement or Appendix B for differential measurement. Any suitable proprietary apparatus may be used and suitable designs are shown in Figure 1.

4.2 Construction materials. For measurements from ambient temperature to 800 °C, transparent fused silica or fused quartz shall be used for construction of the test piece holder. For measurements from ambient temperature to temperatures above 800 °C, an alumina ceramic of at least 99.8 % Al_2O_3 shall be used for construction of the test piece holder.

NOTE For information on the availability of suitable materials, apply to Enquiry Section, BSI, Linford Wood, Milton Keynes MK14 6LE.

The push rod for transmitting the displacement of the test piece to the measuring device shall be of the same material as the test piece holder.

4.3 Test piece holder. Possible constructions of the test piece holder are shown in Figure 2. The outer sleeve shall have an end-plate with a surface ground flat to $\pm 10 \mu\text{m}$. The normal to the surface of the end-plate shall be $\pm 1^\circ$ of the measurement axis.

For test piece holders constructed from fused silica (4.2), the end-plate shall be rigidly fixed to the outer sleeve by flame fusing at a point remote from the end-plate surface. Examples of construction for horizontal and vertical use are shown in Figure 2(a).

For test piece holders constructed from alumina (4.2), fusion is not possible. For vertical measuring apparatus, the outer sleeve shall be fixed to a flat plate using refractory cement outside the joint as shown in Figure 2(b). For non-vertical measuring apparatus in which the sleeve end is free-standing, a closed end alumina tube shall be used, and the end-plate machined to fit inside the tube end as shown in Figure 2(c). The assembly shall then be fixed in position with a suitable refractory cement, and then fired to a temperature greater than 1 400 °C under an axial load.

NOTE The firing under load minimizes the risk of movement of the assembly during use. Some movement may still occur during initial use. Thermal cycling should be continued until the net movement over a thermal cycle to the highest temperature at which the apparatus is to be used is less than 1 μm .

4.4 Test piece mounting. For vertical measuring apparatus, the test pieces shall be free-standing and mechanically stable on the end-plate (4.3). For measuring apparatus which is horizontal or inclined to the horizontal, the sideways movement or twist of the test piece shall be restricted, without any restriction of axial movement, by a suitable arrangement (see note).

NOTE This may be done, for example, by:

- a) using a vee-block cut to fit into the test piece holder;
- b) using test pieces of such dimensions that either a neat sliding fit (but see clause 5) or support on two edges is obtained;
- c) using an arrangement of supporting silica glass balls, as shown in Figure 3.

Where the apparatus is designed for differential measurements, two test pieces shall be suitably mounted parallel to each other, contacting on a single end-plate.

4.5 Thermocouples, type R, complying with BS 4937-2. One thermocouple shall be placed with its junction in contact with the surface of the test piece near its mid-point. Two other thermocouples shall be placed at each end of the test piece, and used in differential mode as shown in Figure 4, to periodically determine the temperature distribution along the test piece.

4.6 Heating or cooling device, comprising a suitable tube furnace or cooling device designed to give a uniform temperature zone of length greater than that of the test piece during the normal thermal cycling of the test (clause 6). The device shall heat or cool the test piece contained in its holder (4.3) and in any surrounding protection tube.

The variation in temperature along the length of the test piece within the device shall be determined using differential thermocouples positioned as shown in Figure 4, and shall not exceed 2 K during thermal cycling.

4.7 Temperature programmer and power control unit, for temperature control of the heating and cooling device (4.6), incorporating a thermocouple (4.5) positioned in the uniform temperature zone of the device. This apparatus shall be such that for tests at a constant rate of change of temperature, deviation from a smooth rate of change shall not exceed ± 1 K, as determined by the thermocouple in contact with the test piece. For tests at a series of steady temperatures, variations in temperature shall not exceed ± 1 K of the mean temperature.

4.8 Expansion measuring device, either a micrometer, dial gauge, linear displacement transducer, or an interferometer.

The device shall have a capability of measuring displacements of 0.001 % of the length of the test piece (see clause 5), and individual measurements shall be repeatable to this accuracy.

Where the apparatus is designed for differential measurements, the measuring device shall directly record the differential movement of the two push rods.

NOTE 1 The difference between separately measured push rod displacements is not acceptable as a measurement.

NOTE 2 Ideally, the expansion measuring device should have its temperature controlled to ± 1 K, using a thermostatic device such as a water jacket operating at a temperature near to, but preferably a little above room temperature. Checks should also be made to ensure that any electronic amplifiers or recording devices used such as a transducer amplifier and recording voltmeter, have outputs that are insensitive to room temperature change. If such changes in output exceed the equivalent of a displacement of 10^{-3} mm for a 10 K change in room temperature, then the room temperature should ideally be controlled to ± 1 K.

4.9 Data recording unit, providing for simultaneous recording of test piece temperature and displacement measuring device output. The sensitivity for temperature measurements shall be 0.1 K.

NOTE This is equivalent to 1 μ V on type R thermocouples (see 4.5).

The thermocouple (4.5) shall have a reference cold junction which may be the temperature of the thermostatically controlled measurement unit, a separate thermostatic unit, or electronic compensation in the recording device as appropriate.

The sensitivity for displacement measurement shall be as described in 4.8.

5 Test pieces

Materials for testing should be sampled in accordance with the guidance given in BS 7134-0.

The dimensions of the test pieces are dependent on the design of the apparatus (clause 4), and in particular the temperature homogeneity of the heating or cooling device (4.6).

The length of the test piece shall be similar to that of any certified reference material used to calibrate the apparatus as in Appendix A. For the differential type of apparatus, the length of the test piece shall be within ± 0.2 mm of the length of the piece of reference material.

The cross section of the test piece may be any suitable shape consistent with the design of the apparatus. For materials of thermal conductivity less than 10 W (m K)^{-1} dimension shall be less than 5 mm.

Ends of the test piece shall be ground to a radius of curvature between 1 mm and 20 mm, so as to ensure point contact with the flat ends of the test piece holder (4.3) and push rod of the measuring device (4.8), except where a vertical measuring device is used with a free-standing test piece, where the lower end of the test piece in contact with the reference base may be ground flat, perpendicular to the test piece axis. In this case the test piece shall be mechanically stable on the reference base.

6 Procedure

Measure the length of the test piece at room temperature, using vernier callipers in accordance with BS 887, a micrometer, or other suitable measuring device, to an accuracy of either:

- a) ± 0.05 mm for lengths of 10 mm or above;
- b) ± 0.02 mm for lengths of below 10 mm.