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Semiconductor devices — Micro-electromechanical devices

Part 11: Test method for coefficients of linear thermal expansion of free-standing materials for micro-electromechanical systems

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The UK participation in its preparation was entrusted to Technical Committee EPL/47, Semiconductors.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Semiconductor devices - Micro-electromechanical devices - Part 11: Test method for coefficients of linear thermal expansion of free-standing materials for micro-electromechanical systems
(IEC 62047-11:2013)

Dispositifs à semiconducteurs -
Dispositifs microélectromécaniques -
Partie 11: Méthode d'essai pour les coefficients de dilatation thermique linéaire des matériaux autonomes pour systèmes microélectromécaniques
(CEI 62047-11:2013)

Halbleiterbauelemente -
Bauelemente der Mikrosystemtechnik -
Teil 11: Prüfverfahren für lineare thermische Ausdehnungskoeffizienten für freistehende Werkstoffe der Mikrosystemtechnik
(IEC 62047-11:2013)

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Foreword

The text of document 47F/154/FDIS, future edition 1 of IEC 62047-11, prepared by IEC/TC 47F "Microelectromechanical systems" of IEC/TC 47 "Semiconductor devices" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 62047-11:2013.

The following dates are fixed:

• latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2014-05-21
• latest date by which the national standards conflicting with the document have to be withdrawn	(dow)	2016-08-21

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Annex ZA
(normative)

Normative references to international publications with their corresponding European publications

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

NOTE When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

Publication	Year	Title	EN/HD	Year
IEC 62047-3	-	Semiconductor devices - Micro-electromechanical devices - Part 3: Thin film standard test piece for tensile-testing	EN 62047-3	-

CONTENTS

SEMICONDUCTOR DEVICES – MICRO-ELECTROMECHANICAL DEVICES –

Part 11: Test method for coefficients of linear thermal expansion of free-standing materials for micro-electromechanical systems

1 Scope

This part of IEC 62047 specifies the test method to measure the linear thermal expansion coefficients (CLTE) of thin free-standing solid (metallic, ceramic, polymeric etc.) micro-electro-mechanical system (MEMS) materials with length between 0,1 mm and 1 mm and width between 10 μm and 1 mm and thickness between 0,1 μm and 1 mm, which are main structural materials used for MEMS, micromachines and others. This test method is applicable for the CLTE measurement in the temperature range from room temperature to 30 % of a material's melting temperature.

2 Normative References

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 62047-3, *Semiconductor devices – Micro-electromechanical devices – Part 3: Thin film standard test piece for tensile-testing*

3 Symbols and designations

Symbols and corresponding designations are given in Table 1.

Table 1 – Symbols and designations

Symbol	Unit	Designation
g	μm	Gauge length
L_0	μm	Initial length of a test piece
L_T	μm	Length of a test piece at temperature T
T	°C	Temperature
t	μm	Thickness of a test piece
w	μm	Width of a test piece
α_{av}	1/°C	Average coefficient of thermal expansion of a test piece
α_S	1/°C	Average coefficient of thermal expansion of a substrate
δ_T	μm	Thermal deformation
ε_T	1	Thermal strain

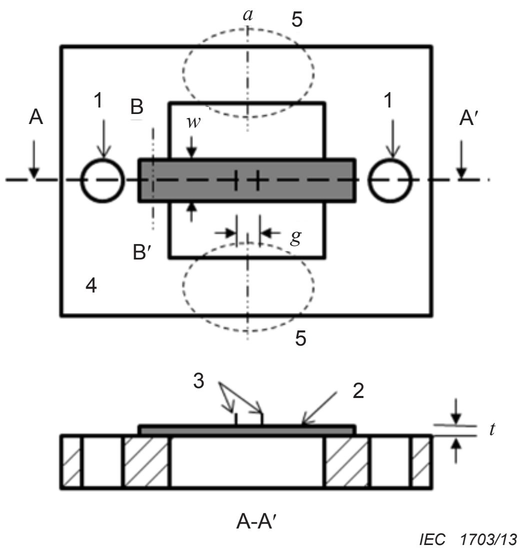
4 Test piece

4.1 General

The test piece shall be prepared in accordance with the IEC 62047-3. It should be fabricated through the same processes used for the device where the thin film is applied. It should have dimensions in the same order of that of the objective device component in order to minimize the size effect. There are many fabrication methods depending on the applications. A typical test piece fabrication method based on MEMS processes is shown in Annex A.

4.2 Shape of test piece

The dimensions of a test piece, such as thickness (t), width (w), and initial length (L_0), in Figure 1 should be designed to be the same order of the device. The dimensions shall be specified within the accuracy range of ± 1 % of the corresponding length scale. The cross sections along the line *A-A′* are indicated as cross-hatching in Figure 1. The gauge length in Figure 1 shall be measured from centre to centre of the gauge marks.



Key

1 holes for die fixing, tying a yarn or wire for the weight hanging

2 free-standing test piece

3 gauge marks to define a gauge length

4 substrate to accommodate a test piece

5 portions to be separated before testing to make a test piece free-standing

NOTE Imaginary line "*a*": The support straps "5" can be separated by cutting those along this line.

Figure 1 – Thin film test piece

4.3 Test piece thickness

Each test piece thickness shall be measured and the thickness should be recorded in the report. Each test piece thickness should be measured directly with calibrated equipment (for example scanning electron microscope, ellipsometer, etc.). However, the film thickness evaluated from step height (by scanning probe microscope, white light interferometric microscope, or surface profilometer, etc.) along the line *B-B′* in Figure 1 can be used as the thickness of a test piece.

4.4 In-plane type test piece

The internal stress of the test piece should have proper values in order not to cause curling of the test piece. Gauge marks should be formed in the middle of a test piece. The gauge marks should not restrict the elongation of the test piece and should have small influence on test result. The stiffness of the gauge mark should be less than ± 1 % of that of the test piece. The symmetry in the thickness direction should be maintained in order to avoid the curling of the test piece. A dummy part shall be attached to a test piece as shown in Figure C.1.

4.5 Out-of-plane type test piece

An out-of-plane type test piece may be used if the free-standing test piece has thickness below 1 μm or has low strength to hang a weight. The holes and gauge marks in Figure 1 are not necessary in case of out-of-plane type test. The supporting straps don't need to be separated. The test piece should be buckled concavely or convexly before measurement.

5 Testing method and test apparatus

5.1 Measurement principle

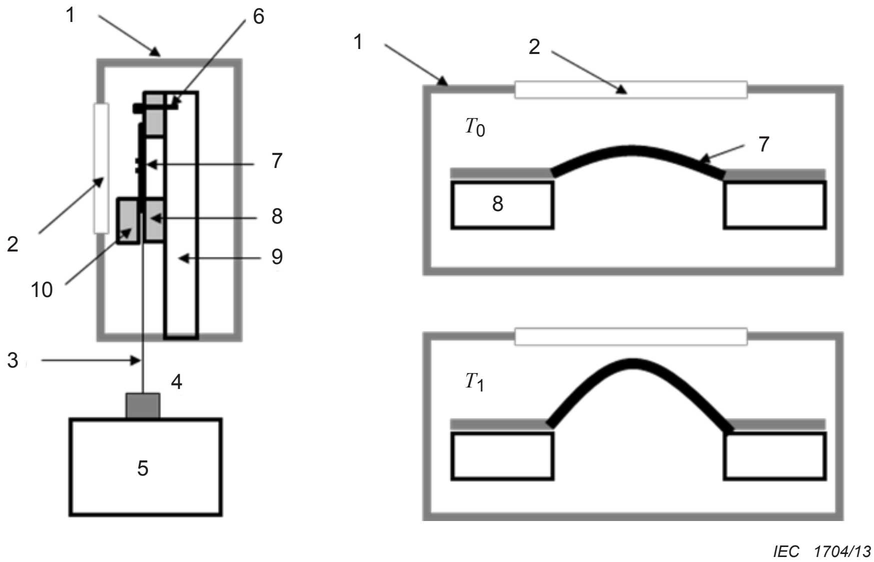
5.1.1 General

The average CLTE value shall be obtained by linearly correlating the thermal strain change ($\Delta\varepsilon_T$) by the corresponding temperature change (ΔT).

$$\alpha_{av} = \frac{\Delta\varepsilon_T}{\Delta T} \qquad (1)$$

The thermal strains shall be obtained with two kinds of test methods as shown in Figure 2.

In-plane test method shall be preferred to out-of-plane method in the view points of accuracy and uncertainties. If there is no test setup as shown in Figure 2 a) and Figure C.1, out-of-plane method shall be used as an alternative because the out-of-plane method needs a furnace and measuring equipment.



a) In-plane type b) Out-of-plane type

Key

1 heating furnace equipped with a hatch
2 viewport to observe and measure deformation of a test piece
3 metal wire or yarn to hang a weight
4 weight
5 translational stage to hold and release a weight
6 bolt to fix a die to the test die holder
7 free-standing test piece
8 test die
9 test die holder
10 dummy part for the symmetry of a test piece

Figure 2 – CLTE measurement principles

5.1.2 In-plane method

The thermal deformation (δ_T) shall be measured directly as a function of temperature by using a noncontact in-plane displacement measurement technique (laser interferometry, 2-D digital image correlation, etc.). The specimen should be in a furnace as shown in Figure 2a). The weight should be hung to a test piece in order to make it flattened. The elastic modulus should be independent of temperature in the range of measurement. The plastic deformation due to weight (yielding) or temperature (creep) should be avoided. The thermal strain shall be calculated by dividing the elongation by the gauge length.

$$\varepsilon_T = \frac{\delta_T}{g} \tag{2}$$

5.1.3 Out-of-plane method

The entire profile of a specimen along the length direction should be measured as a function of temperature by an accurate out-of-plane displacement measurement method (white light interferometric microscope, laser Doppler interferometer, 3-D digital image correlation, etc) as shown in Figure 2b). A test piece should be initially buckled. The initial length (L_0) at room temperature and successive lengths (L_T) at different temperatures of a specimen shall be calculated with the profiles measured. The thermal deformation (δ_T) shall be the difference between L_T and L_0. The thermal strain shall be calculated by dividing the deformation by the initial length.

$$\varepsilon_T = \frac{\delta_T}{L_0} = \frac{L_T - L_0}{L_0} \tag{3}$$

The CLTE of a substrate should be considered to calculate the accurate CLTE of the test piece because both experience the same amount of temperature change. The substrate effect shall be considered by adding the CLTE of the substrate to the average CLTE value from measurement. The CLTE of the substrate should be measured by using a test standard [1, 2, 3][1] if there is no certified CLTE value for the substrate.

$$\alpha_{av} = \frac{\Delta\varepsilon_T}{\Delta T} + \alpha_S \tag{4}$$

5.2 Test apparatus

5.2.1 General

The test piece should be seated in a furnace. The temperature of the furnace should be controlled within ± 1 °C by the feedback control.

5.2.2 In-plane method

A test apparatus shall be equipped with basic components shown in Figure 2a). A transparent window like a glass shall be used as a viewport. The hatch of a furnace should be closed and a predetermined weight should be hung to the yarn or metal wire to make a test piece flat enough but not to the point where it could yield. A test piece should be in a free-standing state before heating it up. See Annexes B and C.

5.2.3 Out-of-plane method

A furnace having a view port is only needed to heat up a test piece. A test piece should be in a free-standing state before heating it up. See Annex D.

5.3 Temperature measurement

The method of temperature measurement should be sufficiently sensitive and reliable. Temperature measurements should be made with a calibrated thermometer. Contact (thermocouple, etc.) or noncontact (infrared thermometers, optical pyrometers, etc.) thermometers shall be used. The temperature sensor that enables to measure ± 0,5 % of the maximum temperature accuracy shall be used and should be calibrated periodically. The temperature sensing points should be located very near to a test piece to measure the temperature accurately. The temperature distribution in the length direction should be doubly checked by a noncontact sensor like an IR thermometer.

5.4 In-plane test piece handling

A metal wire or yarn should be tied around a right hole in Figure 1 for the later weight hanging. The supporting portions in Figure 1 should be separated by cutting those before setting it up to the furnace. The test piece should be handled with special care after separating the supporting portions. This step can be skipped if a test piece is robust enough to handle easily. See Annex B.

---

1 Figures in square brackets refer to the bibliography.

5.5 Thermal strain measurement

A displacement measurement method that enables to measure 0,01 % strain value shall be used. Displacement should be measured at every 1 °C during a test to adequately define the temperature-strain curve.

5.6 Heating speed

The thermal strains should be recorded as a function of temperature while raising the temperature below the rate of 1 °C/min to avoid thermal inertia.

5.7 Data analysis

5.7.1 General

The average CLTE shall be calculated by using one of the following methods.

5.7.2 Terminal-based calculation

The average linear CLTE value shall be calculated by dividing the thermal strain difference ($\Delta\varepsilon_T$) by the corresponding temperature difference (ΔT). The temperature-strain curve should be linear in the range of interest.

5.7.3 Slope calculation by linear least squares method

The linear least squares method shall be used to fit the thermal strain (ε_T) versus temperature (T) data. The average CLTE (α_{av}) shall be the slope of the linearly fitted curve. The intercept on the thermal strain axis (ε_{T0}) does not affect the result at all. The coefficient of correlation shall be over 0,95 to ensure the linearity. See Annexes E and F.

$$\varepsilon_T = \alpha_{av} T + \varepsilon_0 \tag{5}$$

6 Test report

The test report shall contain at least the following information.

a) reference to this international standard;
b) identification number of the test piece;
c) displacement measuring equipment;
   – type;
   – sensitivity and accuracy;
d) test piece material;
   – in case of single crystal: crystallographic orientation;
   – in case of polycrystal: texture and grain size;
e) shape and dimension of test piece;
   – type (in-plane or out-of-plane)
   – picture;
   – gauge length (in-plane method only);
   – thickness;
   – width;
f) test piece fabrication method and its detail;
   – deposition method;

  - fabrication condition;

g) weights and stresses induced (in-plane method only);

h) temperature measurement method and its accuracy;

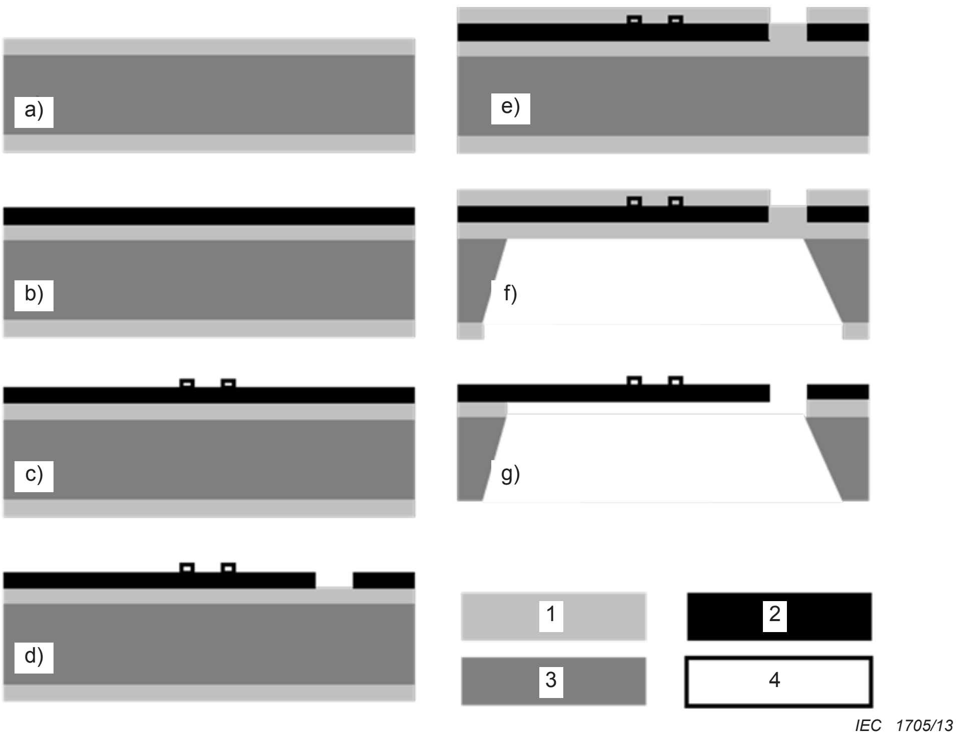
i) measured properties and results;
  - thermal strain curve;
  - average linear coefficient of thermal expansion;
  - calculation methods (terminal-based or least squares method);
  - temperature range.

Annex A
(informative)

Test piece fabrication

A test piece should be fabricated using the same MEMS processes as those of the device where the thin film is applied. A typical test piece fabrication process is shown in Figure A.1.

a) Deposit oxide layers on both sides of a bare substrate like a (100) silicon wafer.
b) Deposit test material (for example, Al, Au, Si_3N_4, etc.) on top of the oxide film. An adhesion layer shall be deposited between oxide and test material layers to improve adhesion between them. The thickness of the adhesion layer should be minimized in order not to affect the measurement.
c) Deposit and pattern a thin layer to form gauge marks. This process shall be skipped according to the displacement measurement techniques. The thickness should be minimized in order not to reinforce the test piece.
d) Pattern the target film to make the shape of a test piece. The patterning is done by a photolithography process.
e) Passivate the patterned test piece by oxide or photoresist.
f) Etch the substrate from backside to make the film free-standing.
g) Remove the photoresist and oxide to get a free-standing test piece.



Key

1 silicon dioxide, SiO_2
2 test piece material
3 substrate
4 markers to form the gauge length

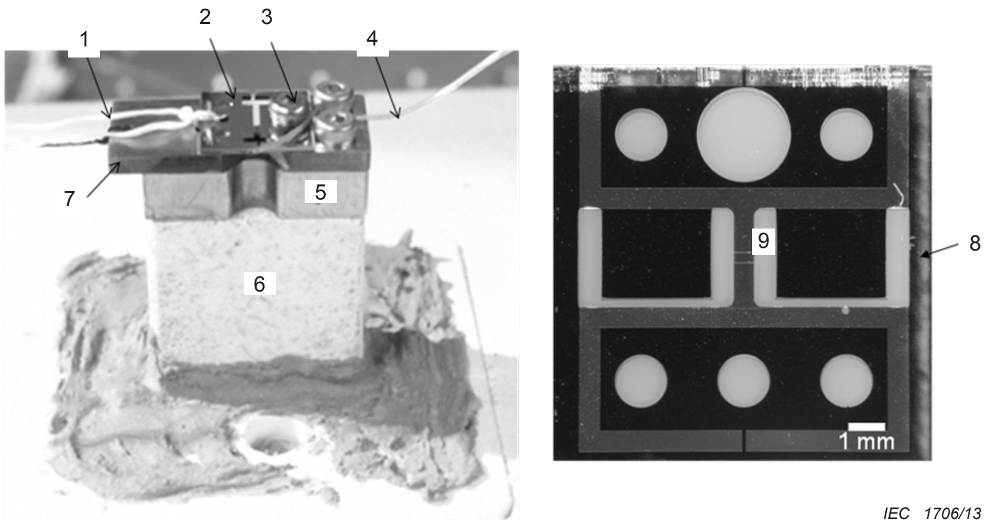
NOTE The fabrication processes depend on the measurement methods and applications.

Figure A.1 – Schematic test piece fabrication process

Annex B
(informative)

Test piece handling example

A metal wire or yarn (1) is tied around a lower centre hole of a test piece (See Figure B.1) in order to subsequently hang a weight. A test die (2) should be fixed to a base jig (5) with the aid of a safety jig (7), a bolt (3) and wax, which remains solid at room temperature but melts at a certain melting temperature of approximately 60 °C. The two support straps (8) should be cut with a diamond saw to leave a completely free-standing uniaxial test piece (9). This set is assembled to the furnace jig (6) as shown in Figure B.1. A thermocouple (4) is placed very close to a test piece to measure the temperature accurately.



Key

1 yarn
2 test die
3 bolt
4 thermocouple
5 base jig
6 furnace jig
7 safety jig
8 support strap
9 free-standing test piece

Figure B.1 – Auxiliary jigs and a specimen example

Annex C
(informative)

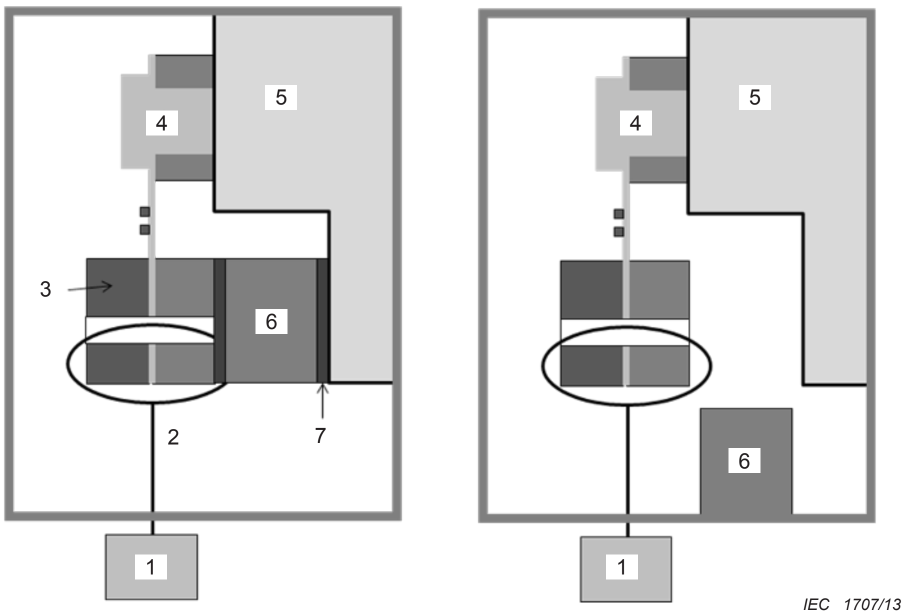
Test piece releasing process

The test piece releasing process is schematically illustrated in Figure C.1.

a) Set up the whole assembly containing test die, base jig, safety jig and furnace jig in a heating furnace. Attach a balancing dummy part to a test die to make the free-standing test piece symmetric in the thickness direction. See Annex B.

b) Hang a weight to the yarn.

c) Raise the furnace temperature around 60 °C to melt the wax among the test die, safety jig, and base jig. After melting the wax, the test piece becomes free-standing carrying a weight.



Key

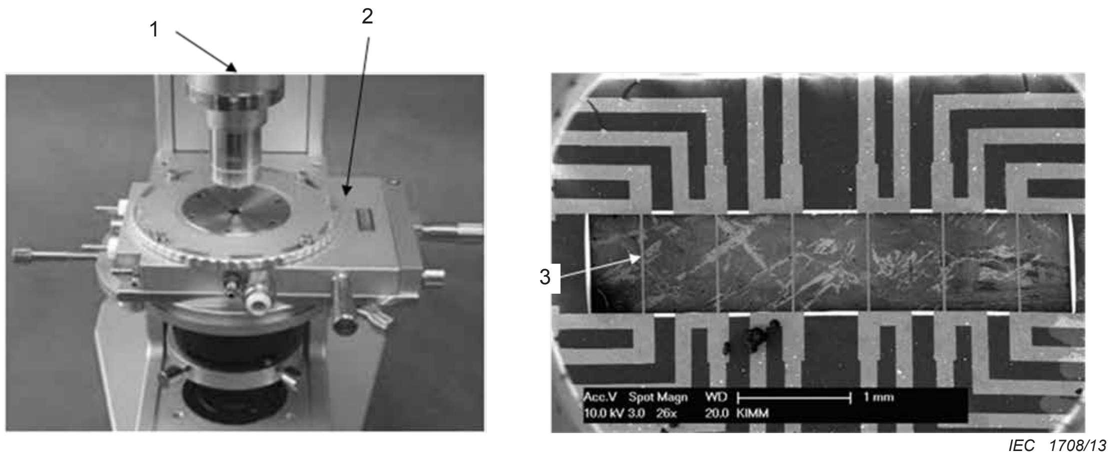
1 weight
2 yarn
3 balancing dummy part
4 bolt
5 base jig
6 safety jig
7 wax

Figure C.1 – Schematic illustration showing the test piece releasing process

Annex D
(informative)

Out-of-plane test setup and test piece example

Figure D.1 presents examples of a test setup and a test piece for the out-of-plane test method. The test piece is initially buckled in order to measure the thermal strain from the beginning. The status of a test piece is checked by measuring its profile with a noncontact out-of-plane displacement measuring equipment.



Key

1 white light interferometric microscope

2 heating furnace

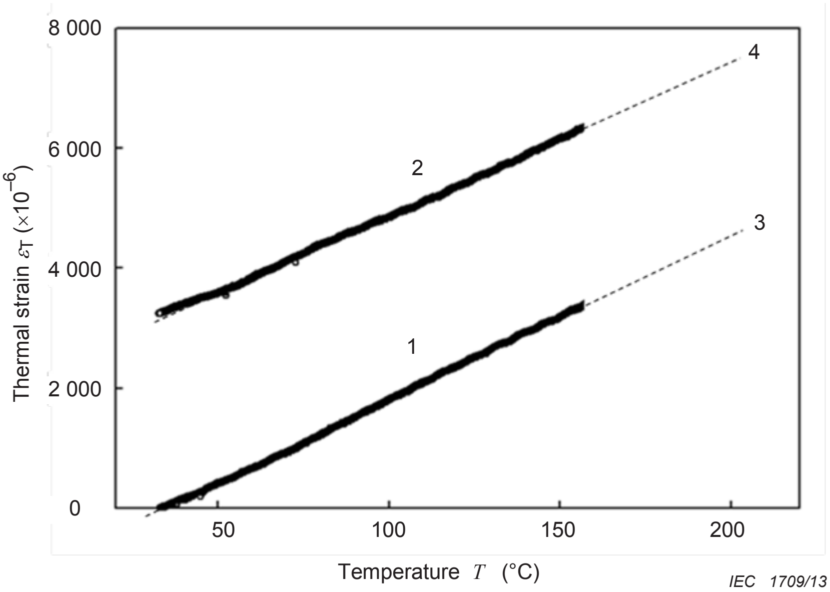
3 free-standing test piece (20 μm wide and 1 mm long, gold)

Figure D.1 – Example of test setup and test piece

Annex E
(informative)

Data analysis example in in-plane test method

Figure E.1 presents the result of an in-plane measurement in which the aluminium test piece was heated from room temperature of 25 °C to 160 °C and then cooled back to room temperature. The two curves were shifted on purpose to see the differences in more detail. The test had a weight of 20 grams (74 MPa stress). The average CLTE value was estimated as the slopes of the thermal strain versus temperature curves. The CLTE was estimated as 28×10^{-6}/°C in the heating stage and 25×10^{-6}/°C in the cooling stage.



Key

1 data in the heating stage

2 data in the cooling stage

3 line fitted by linear least squares analysis for the data in the heating stage

4 line fitted by linear least squares analysis for the data in the cooling stage

Figure E.1 – Example of CLTE measurement with an aluminium test piece

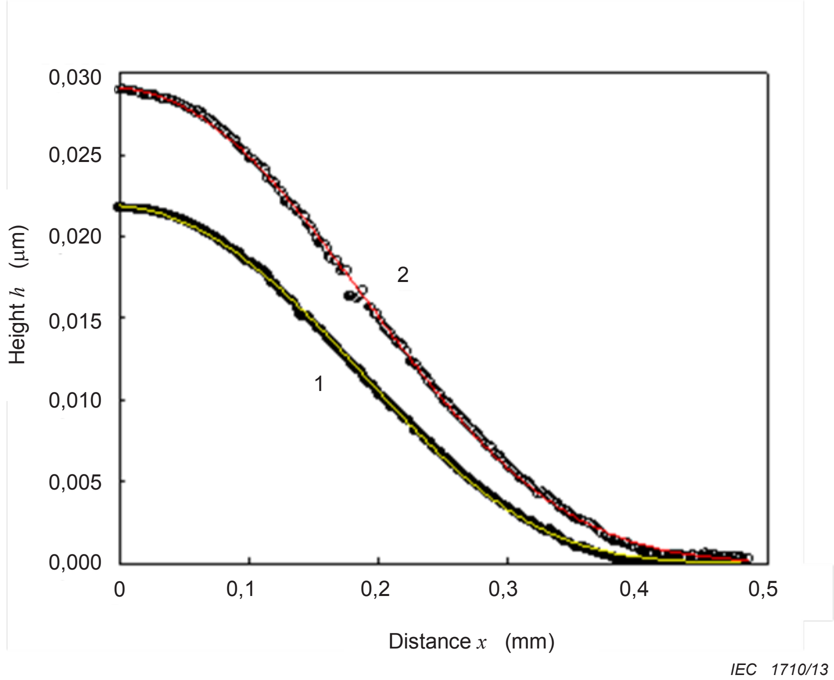
Annex F
(informative)

Data analysis example in out-of-plane test method

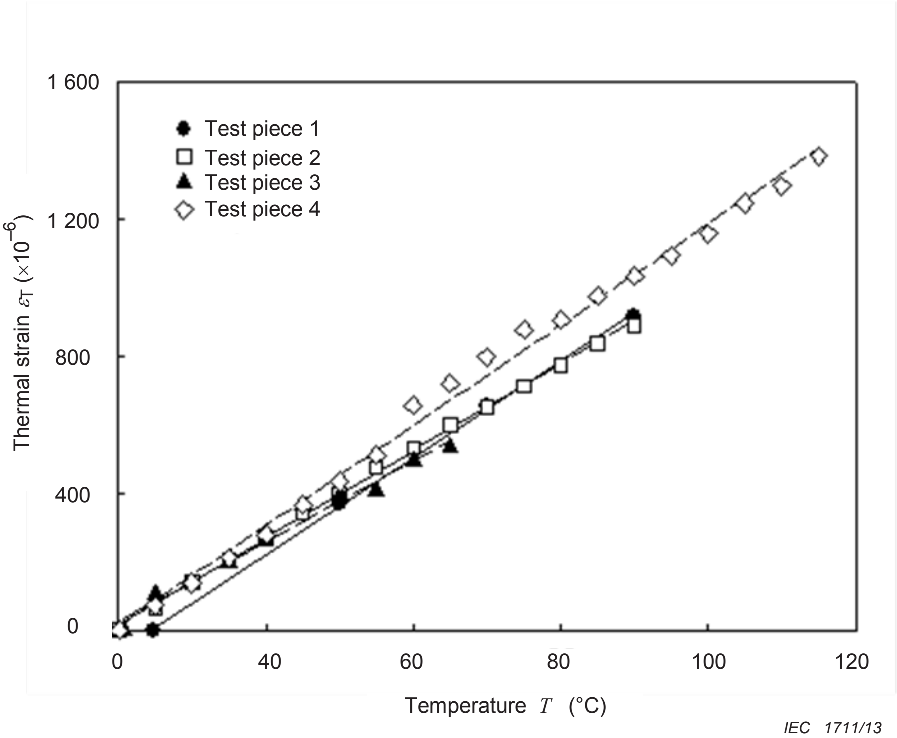
Figure F.1a) presents two examples of profiles measured by a white light interferometric microscope for gold test piece at two different temperatures ($T_2 > T_1$). The data points should be fitted to get a closed form equation to integrate and thus calculate the length. In principle, the data is fitted to sinusoidal equation because it is the solution to the buckling problem. However, the tail portions in Figure F.1 converge to 0 because the test piece was fixed to the substrate. A four-parameter (a, b, c, x_0) Weibull curve as shown in Equation (F.1) is one of the appropriate curve fitting models. The fitted curves are shown in Figure F.1a) with their raw data points. The data points follow the curve very well.

$$y(x) = a\left(\frac{c-1}{c}\right)^{\frac{1-c}{c}}\left[\frac{x-x_0}{b}+\left(\frac{c-1}{c}\right)^{\frac{1}{c}}\right]^{c-1} e^{-\left[\frac{x-x_0}{b}+\left(\frac{c-1}{c}\right)^{\frac{1}{c}}\right]} + \frac{c-1}{c} \qquad \text{(F.1)}$$

The thermal strains for four different specimens were calculated by Equation (3) and plotted in Figure F.1b) while raising the temperature from room temperature of 20 °C to 120 °C. The symbols represent data points and the lines fitted by linear least-squares method. The average CLTE value was estimated as the slopes of the thermal strain versus temperature curves as explained in 5.7.3. The CLTE was estimated to 13,3 × 10^{-6}/°C. The final CLTE shall be calculated by adding the CLTE of the silicon substrate of 3 × 10^{-6}/°C. The final CLTE of the gold film is 16,3 × 10^{-6}/°C.



a) Out-of-plane profiles at two temperatures



b) Thermal strain as a function of temperature

Key

1 data and four-parameter Weibull fitting at temperature T_1

2 data and four-parameter Weibull fitting at temperature T_2 ($> T_1$)

Figure F.1 – Example of CLTE measurement with a gold test piece

Bibliography

[1] ASTM E228 – 11, *Standard Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer*

[2] ASTM E289 – 04(2010), *Standard Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry*

[3] ASTM E831 – 06, *Standard Test Method for Linear Thermal Expansion of Solid Materials by Thermomechanical Analysis*

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