The Institute for Interconnecting and Packaging Electronic Circuits
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IPC-TM-650
TEST METHODS MANUAL

Number	
2.4.41.1	
Subject	
Coefficient of Thermal Expansion by the Vitreous Silica (Quartz) Dilatometer Method	
Date	Revision
8/97	**A**
Originating Task Group	
N/A	

1.0 Scope

1.1 To describe the vitreous silica dilatometer method for determining the linear thermal expansion of laminated materials within the temperature range of –55°C to 100°C. Inorganic substrates (non-laminated) shall be tested within a range of –55° to 150°C.

2.0 Applicable Documents

ASTM-E-228 Standard Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer

ASTM-D-696 Test for Coefficient of Linear Thermal Expansion of Plastics

ASTM-E-831 Test for Linear Thermal Expansion of Solid Materials by Thermodilatometry

ASTM-E-77 Verification and Calibration of Liquid-in-Glass Thermometers

ASTM-E-220 Calibration of Thermocouples by Comparison Techniques

ASTM-E-644 Testing Industrial Resistance Thermometers

3.0 Test Specimen

3.1 Laminated materials which may or may not contain metal layers.

3.2 Nominal test specimen dimensions shall be 1/4 inch wide x 2 inch –4 inch long x 1/8 inch minimum thickness. End surfaces shall be ground parallel. Any deviation from nominal should recognize thermal gradients of the temperature chamber, thermal lag of specimen and any bending of specimen. Thicknesses under 1/8 inch shall be supported by adequate clamping devices unless it is certain that the specimen will remain straight during testing.

4.0 Apparatus

4.1 Vitreous silica dilatometer of either the tube or push rod type to determine the change in length of a solid material as a function of temperature. The temperature is controlled at a constant heating or cooling rate. The linear thermal expansion and the coefficients of linear thermal expansion (CTE) are calculated from the recorded data.

This device measures the difference in thermal expansion between a test specimen and the vitreous silica parts of the dilatometer (Figure 1).

4.2 Specimen holder (tube) and probe shall be made of vitreous silica. The probe contact shall be flat or be rounded to approximately a 10 mm radius.

4.3 Chamber for uniformly heating and cooling the specimen. The specimen temperature change rate shall be controlled. The temperature gradient in the specimen shall not exceed 0.5°C/cm.

4.4 Transducer, for measuring the difference in length between the specimen and the specimen holder with an accuracy of at least ± 0.5µm. The transducer shall be protected or mounted so that temperature changes will not affect the readings by more than 1.0µm.

4.5 Micrometer, for measuring the reference length, L_o, of the specimen with an accuracy of at least ± 25µm.

4.6 Thermocouple, types E, K, or T, for measurement of the specimen temperature. (Type E is NiCr versus constantan, type K is NiCr versus NiAl and Type T is Cu versus constantan.)

4.7 Recorder or data logger for collecting temperatures and lengths.

5.0 Procedure

5.1 Sample Preparation Rough cut with a band saw or metallurgical cut-off wheel and finish machining by grinding. Care must be exercised to remove roughness from specimen ends. The ends shall be parallel to ± .001 inch/inch.

5.2 Sample condition (only for laminated, organic specimens).

5.2.1 The specimen shall be immersed in isopropyl alcohol and agitated for twenty seconds.

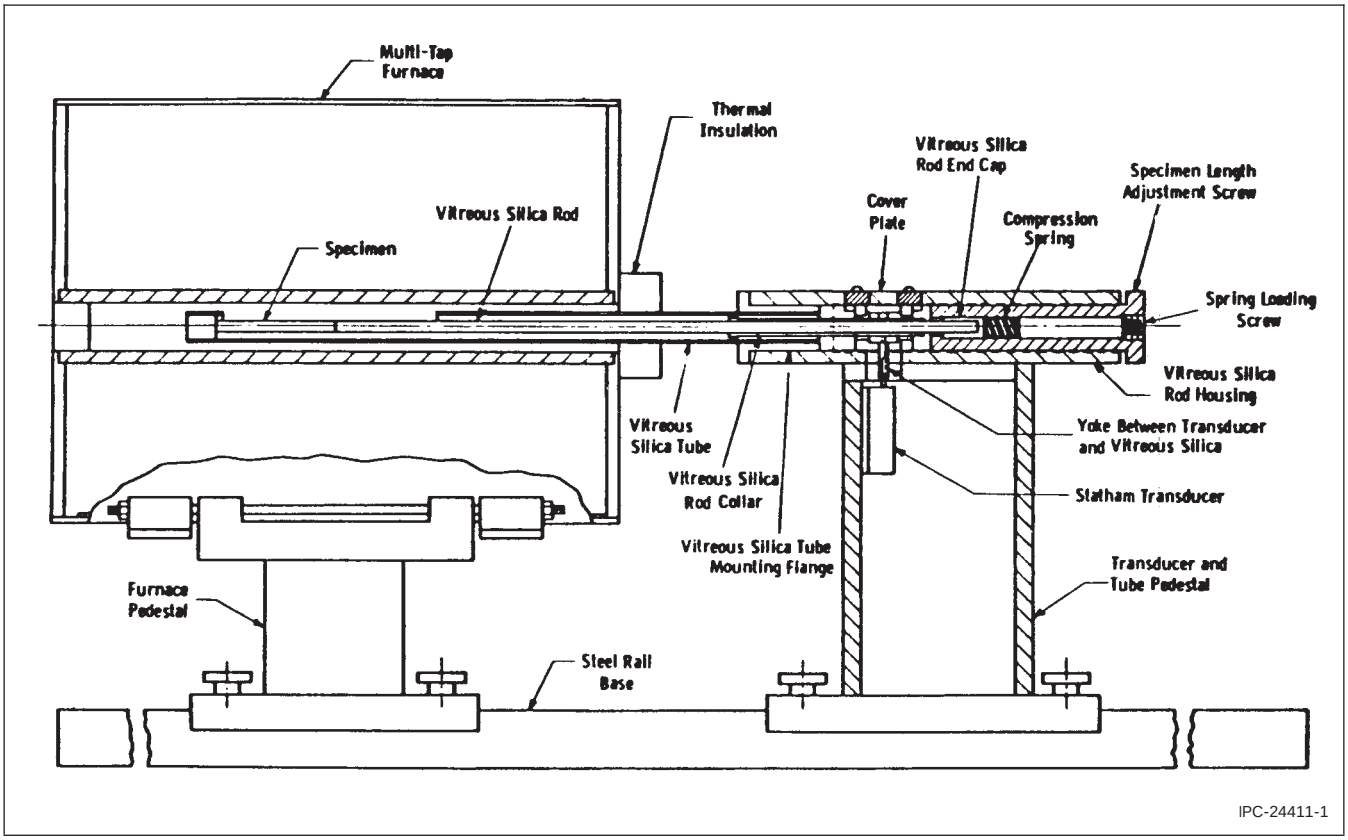

Figure 1 Cutaway view of vitreous silica tube dilatometer

5.2.2 Condition E-1/110.

5.2.3 Condition C_1-40/23/50.

5.3 Calibration

5.3.1 The transducer shall be calibrated by imposing a series of known displacements with a precision screw micrometer or set of end gage blocks.

5.3.2 The temperature sensor shall be calibrated according to an appropriate ASTM method (E-220) or procedure recommended by the National Bureau of Standards.

5.3.3 The dilatometer, as a total system, shall be calibrated by measuring two reference materials of known thermal expansion. One of the materials should have an expansion close to the sample specimen, and the other close to that of the dilatometer.

5.3.4 Recommended standard reference materials:

- NBS Fused Silica – SRM 739; CTE ~ .55 PPM/°C (for calibration of dilatometer)
- NBS Single Crystal Sapphire - SRM 732; CTE ~ 5.5 PPM/°C (for use with ''low expansion'' materials)
- OFHC Copper; CTE ~ 17.3 PPM/°C (for use with ''high expansion'' materials)

5.3.5 The expansion of the dilatometer system, $(\Delta L/L_o)_s$, and the calibration constant, for corrections of lead lag, temperatures, etc., are determined at 20°C intervals using the following equations:

$$(\Delta L/L_o)_s = (\Delta L/L_o)_t - (\Delta L/L_o)_m$$

$$A = \frac{\left(\frac{\Delta L}{L_o}\right)_t - \left(\frac{\Delta L}{L_o}\right)_s}{\left(\frac{\Delta L}{L_o}\right)_m}$$

where:

L_o = specimen length

$\left(\frac{\Delta L}{L_o}\right)_t$ = certified expansion of the reference material.

$(\Delta L/L_o)_m$ = the measured expansion of the reference material.

$\left(\frac{\Delta L}{L_o}\right)_s$ = the expansion of the vitreous silica parts of the dilatometer.

5.4 Test Procedure Following the conditioning steps per 5.2, two thermal cycles shall be conducted per test. The first is to normalize the specimen and the second to generate data for the calculation of CTE.

5.4.1 Measure the initial length of the specimen, using the micrometer to ± .001 inch.

5.4.2 Place the specimen in the dilatometer after making certain that all contacting surfaces are free of foreign material. Specimens with thickness 0.125 inch shall be supported with side plates. Care must be taken to assure good seating of the specimen against the bottom of the tube bottom and the push rod.

5.4.3 Place the thermocouple sensor in intimate contact with the specimen at midlength.

5:4.4 Mount the transducer to provide a stable contact with the probe. The sample loading force shall be the minimum necessary for proper contact between the rod and specimen, and the bottom of the tube and specimen. Set the transducer at a nominal initial reading.

5.4.5 Place the assembled dilatometer into the chamber and allow the temperature of the specimen to come to equilibrium.

5.4.6 Record the initial readings of the thermocouple and the transducer.

5.4.7 Heat and cool at a constant rate of 2°C/min.

5.4.8 Record length changes as a function of temperature.

5.4.9 Remove the specimen from the fixture and repeat the procedure per 5.4.1-5.4.8, following the first cycle. Remeasurement of the specimen length must not be omitted prior to start of the second cycle.

5.4.10 Test a total of four specimens, two prepared with the length in the machine direction of the laminate reinforcement and two cut in the transverse direction. This quantity is intended to represent the expansion characteristics of a 18 inch x 24 inch panel size.

6.0 Calculations

6.1 Linear thermal expansion (LTE), the change in length per unit length resulting from a temperature change is represented by:

$$\frac{\Delta L}{L_o} = A\left(\frac{\Delta L}{L_o}\right)_a + \left(\frac{\Delta L}{L_o}\right)_s$$

where:

$\left(\frac{\Delta L}{L_o}\right)_a$

is the expansion as indicated by the transducer, ΔL is the observed change in length ($\Delta L = L_2 - L_1$). LTE is often expressed in µm/m (parts per million).

6.2 Mean coefficient of linear thermal expansion – the linear thermal expansion per change in temperature. Represented by:

$$\propto m = \frac{\Delta L/L_o}{\Delta T} = \frac{(L_2 - L_1)}{L_o(T_2 - T_1)}$$

where L_1 and L_2 are the lengths of the specimen at the test temperatures T_1 and T_2.

6.3 Instantaneous coefficient of linear thermal expansion – the slope of the linear thermal expansion curve at temperature T. Represented by:

$$\propto T = \frac{1}{L_o}\frac{dL}{dT,}$$

6.4 Plots of the following are commonly used as required:

$\frac{\Delta L}{L_o}$ vs. T; $\propto$ m vs. T

When reporting the mean coefficient of thermal expansion, the temperature ranges must be specified.