Designation: C1300 – 95 (Reapproved 2020)

Standard Test Method for Linear Thermal Expansion of Glaze Frits and Ceramic Whiteware Materials by Interferometric Method[1]

This standard is issued under the fixed designation C1300; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the interferometric determination of linear thermal expansion of premelted glaze frits and fired ceramic whiteware materials at temperatures lower than 1000 °C (1830 °F).

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E289 Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry

3. Significance and Use

3.1 This test method defines the thermal expansion of glaze frits by the interferometric method. This determination is critical in avoiding crazing (cracking) of these glass coatings due to mismatching of the thermal expansion between the coating and substrate materials.

4. Apparatus

4.1 *Sample Preparation Equipment:*[3,4]

4.1.1 *Glazed Porcelain Crucible,* No. 0.

4.1.2 *Fireclay Crucible,* 102 mm (4 in.) in diameter.

4.1.3 *Rotating Abrasive Grinding Wheel* (a silicon carbide type is satisfactory).

4.2 *Micrometer Calipers,* having a sensitivity such that the index can be read to 0.002 mm (0.0001 in.).

4.3 *Measuring Apparatus,* consisting of fused silica interferometer plates, viewing apparatus, an electric furnace and control, potentiometer, pyrometer, and a suitable monochromatic light source of known wavelength.

4.3.1 *Furnace*—The furnace shall be a vertical electric tube furnace controlled by rheostat or other means so that the heating rate of the furnace can be readily duplicated from room temperature to 1000 °C (1830 °F). The heating rate shall not exceed 3 °C (5.5 °F)/min.

4.3.2 *Temperature-measuring Instrument*—A calibrated platinum versus platinum-rhodium thermocouple (or a Chromel versus Alumel thermocouple if it is frequently calibrated) in conjunction with a potentiometer shall be used. The potentiometer shall be capable of being read to 2 °C (or 4 °F) and shall have automatic compensation for the temperature of the reference junction, or the reference junction shall be held at 0 °C (32 °F) by means of an ice bath.

5. Test Specimens

5.1 For frit samples, three test specimens shall be prepared as follows:

5.1.1 Fill a No. 0 glazed porcelain crucible with frit, place the filled crucible inside a 102 mm (4-in.) diameter fireclay

[1] This test method is under the jurisdiction of ASTM Committee C21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.03 on Methods for Whitewares and Environmental Concerns.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] An example of suitable test equipment and an inferometric method may be found in the paper by Merritt, G. E., "The Interference Method of Measuring Thermal Expansion," *Journal of Research - National Bureau of Standards RP515,* Vol 10, No. 1, January 1933, p. 59.

[4] A description of a permissible automatic fringe recording device may be found in the paper by Saunders, J. B., "An Apparatus for Photographing Interference Phenomenon," *Journal of Research - National Bureau of Standards RP1668,* Vol 35, No. 3, September 1945, p. 157.

crucible partly filled with silica, and work the small crucible down into the silica until approximately 75 % of the small crucible is below the level of the silica.

5.1.2 Place the crucible assembly into a furnace that is at a temperature high enough to just melt the mass. Hold for 15 min after the frit has reached the furnace temperature.

5.1.3 Remove the crucible, rapidly transfer it to another furnace that is at the frit firing temperature, and cool in the furnace at a rate not to exceed 60 °C (110 °F)/h.

5.1.4 Break the small crucible open and break up the vitreous mass. Select six fragments from the interior of the mass (to avoid side portions diluted by the ceramic crucible) having minimum conical dimensions of 3 mm (⅛ in.) at the base and 6 mm (¼ in.) high.

5.2 For fired samples, break and select six samples having minimum conical dimensions of 3 mm (⅛ in.) at the base and 6 mm (¼ in.) in height. For all samples, grind the base of the (cones) flat and cement the flat cone base to the flat end of a glass rod with heated sealing wax. Grind the piece to a finished cone by rotating the rod while the piece is held against a rotating abrasive wheel (a silicon carbide type is satisfactory).

5.2.1 When a reasonably symmetrical cone with a rounded tip is obtained, remove it from the rod by heating the wax or by pressure with the fingertips. Remove all sealing wax with a knife blade or abrasive paper.

5.2.2 The test cone height may be of the order of 4.8 mm (3⁄16 in.). These bases must be smooth and flat. Use No. 0 metallurgical paper to approach the desired figure and then use successively finer papers until the final reduction is made with a No. 3/0 paper.

6. Calibration of Furnace[5]

6.1 Using the following procedure, calibrate the furnace controls to obtain a heating rate of 3 °C (5.5 °F)/min:

6.1.1 Prepare three conical spacers closely approximating the dimensions of the final test pieces described in Section 5. These spacers shall be ground from fragments of refractory ceramic known to have a softening temperature in excess of 1000 °C (1830 °F).

6.1.2 Assemble the upper and lower interferometer plates with three refractory spacers as described in Section 7, except fringe development is not necessary. Place this assembly in the furnace test location. Center the hot junction of an 18- or 20-gauge thermocouple within the triangle formed by the spacers. It will usually be necessary to extend the thermocouple out through the top of the furnace tube. This thermocouple temperature measurement equipment shall meet the requirements in 4.3.2.

6.1.3 The output of this thermocouple shall be used to establish corrections required in calibrating the furnace temperature measuring system. Both temperature values and heating rates shall be so corrected if differences exist.

7. Procedure

7.1 Assemble (outside the furnace) the three test pieces prepared as described in Section 5 between the two interferometer plates as follows:

7.1.1 Place the plate with the one frosted side down within the refractory specimen crucible.

7.1.2 Place the three test pieces on this plate in an equilateral triangle.

7.1.3 Lower the clear plate onto the test pieces keeping the mark or notch identifying the “wedge side” in the “up” position.

7.1.4 Set this assembly at a height comparable to that to be used inside the furnace.

7.2 Rotate the telescope and center it over the test specimen assembly. Direct the monochromatic light source down the tube. If four to eight fringes are present, the setup is correct. If fewer or more fringes are present, adjust the cone heights. In some cases, mere tapping of the specimen assembly will produce the correct number of fringes. Carefully measure and record the height of each cone. Upon achieving the proper number of fringes, place the refractory ring cover on the crucible and recheck for fringes.

7.3 Without rotating the crucible, gently lower it into the furnace and onto the bottom support so that the thermocouple rests at the bottom of the crucible. Cover the top of the furnace with a quartz plate.

7.4 Rotate the telescope and check the fringe pattern. If excessive glare or poor contrast are present, adjust by moving the quartz cover, moving the light source, or releveling the telescope.

NOTE 1—Removal of the telescope eyepiece should reveal a bright dot, which is the true image. This must be in the field or no fringes will be seen. If this bright dot of the true image is not seen when the eyepiece is removed, a great deal of trial and error adjustment of the telescope tripod must be made. A number of false images may also be present. These must be sorted out by inserting the eyepiece and checking to see if fringes are present. If no fringes are seen, the bright dot is a false image.

7.5 Standardize the potentiometer if necessary and set the potentiometer or other temperature-measuring instrument to 38 °C (100 °F).

7.6 Slowly heat the furnace to 38 °C (100 °F). Center the cross hair of the telescope upon any convenient fringe and record the temperature corresponding to each fifth fringe.

7.7 Continue heating the furnace to maintain a 3 °C (5.5 °F)/min temperature rise or less. Below 100 °C a heating rate not exceeding 1.5 °C/min is preferred. For frit samples, when the softening temperature has been reached, as shown by the fringes retreating for at least one fringe, immediately turn off the furnace to avoid reaction with the quartz plates.

[5] Saunders, J. B., “Improved Interferometric Procedure with Application to Expansion Measurements,” *Journal of Research - National Bureau of Standards RP 1227,* Vol 23, No. 1, July 1939, p. 179.

8. Calculations

8.1 Calculate the percentage of linear thermal expansion for each reading as follows:

$$L = (n\lambda/200h) + A_c \quad (1)$$

where:

L = linear thermal expansion, % from starting temperature, t_0 °C, to temperature of observation, t °C,
n = number of fringes passing the reference point during the change from temperature t_0 to temperature t,
λ = wavelength of the light source, μm,
h = height of the specimen at temperature t_0, cm, and
A_c = air correction from temperature t_0 to temperature t, % (see Table 1).

8.2 Prepare a curve by plotting each temperature reading, t, on the horizontal axis against the corresponding percentage expansion along the vertical axis. (See Table 1.)

8.3 Calculate the mean coefficient of thermal expansion, E, for any temperature range, t_2 to t_3 °C, within the limits of the test, as follows:

$$E = L'/[100(t_3 - t_2)] \quad (2)$$

where:

L' = linear thermal expansion, from temperature t_2 °C to temperature t_3 °C as determined from the curve prepared in accordance with 8.2, %,
t_2 = lower temperature in range t_2 to t_3, and
t_3 = higher temperature in range t_2 to t_3.

9. Report

9.1 The report shall include the following:

9.1.1 Designation of material tested,

9.1.2 Method of preparation of test specimen, cooling rate, and so forth,

9.1.3 Identification of type of apparatus used,

9.1.4 Data sheet showing:

9.1.4.1 Form and height of test specimens,

9.1.4.2 Wavelength of light source,

9.1.4.3 Starting temperature,

9.1.4.4 Corrected temperature at each reading,

9.1.4.5 Number of fringes, n, at each reading,

9.1.4.6 Calculation, $n\lambda/200h$, for each reading,

9.1.4.7 Air correction, A_c, for each reading,

9.1.4.8 Percentage of expansion, L, computed for each reading,

9.1.5 The curve (see 8.2) showing temperature plotted against percentage of expansion, and

9.1.6 Mean coefficient of linear thermal expansion per degree Celsius over the desired temperature ranges.

10. Precision and Bias

10.1 The precision and bias of this test method of measuring the linear thermal expansion of glaze frits are as specified in Test Method E289.