Designation: C 539 – 84 (Reapproved 2006)

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

This standard is issued under the fixed designation C 539; 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 frits (porcelain enamel and glaze) 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 289 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 porcelain enamel and 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]

4.1.1 *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 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 (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 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 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.

---

[1] This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.

Current edition approved April 1, 2006. Published April 2006. Originally approved in 1964. Last previous edition approved in 2000 as C 539 – 84 (2000).

[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 Institute of Standards and Technology, Vol 10, No. 1, January 1933, p. 59 (*RP 515*).

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 Institute for Standards and Technology, Vol 35, No. 3, September 1945, p. 157 (*RP 1668*).

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Copyright by ASTM Int'l (all rights reserved); Thu Apr 16 07:34:20 EDT 2009
Downloaded/printed by
Laurentian University pursuant to License Agreement. No further reproductions authorized.

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 flat cones 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 [4]

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-gage 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 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 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.

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.

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

Copyright by ASTM Int'l (all rights reserved); Thu Apr 16 07:34:20 EDT 2009
Downloaded/printed by
Laurentian University pursuant to License Agreement. No further reproductions authorized.

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 7.2, %,
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 Report the following:

9.1.1 Designation of material tested,

9.1.2 Method of preparation of test specimen, cooling rate, etc.,

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 porcelain enamel and glaze frits are as specified in Test Method E 289.

TABLE 1 Air Corrections From 20°C To Temperatures Indicated[A]

Atmospheric pressure, 760 mm Hg.

Temperature, °C	Air Correction, %	Temperature, °C	Air Correction, %	Temperature, °C	Air Correction, %	Temperature, °C	Air Correction, %
20	0.0000	84	0.0049	175	0.0094	320	0.0138
21	0.0001	86	0.0050	178	0.0095	326	0.0139
22	0.0002	88	0.0051			331	0.0140
23	0.0003	89	0.0052	180	0.0096	335	0.0141
24	0.0004			183	0.0097		
25	0.0005	90	0.0053	185	0.0098	340	0.0142
26	0.0006	92	0.0054	188	0.0099	343	0.0143
27	0.0007	94	0.0055			350	0.0144
28	0.0008	96	0.0056	190	0.0100	354	0.0145
29	0.0009	97	0.0057	194	0.0101	359	0.0146
		98	0.0058	197	0.0102		
30	0.0010					364	0.0147
32	0.0011	102	0.0059	200	0.0103	369	0.0148
33	0.0012	103	0.0060	202	0.0104	374	0.0149
35	0.0013	104	0.0061	205	0.0105	378	0.0150
36	0.0014	106	0.0062	208	0.0106		
37	0.0015	108	0.0063			386	0.0151
38	0.0016			210	0.0107	392	0.0152
				214	0.0108	397	0.0153
40	0.0017	110	0.0064	217	0.0109		
41	0.0018	112	0.0065			403	0.0154
42	0.0019	114	0.0066	220	0.0110	409	0.0155
43	0.0020	116	0.0067	223	0.0111	415	0.0156
45	0.0021	118	0.0068	226	0.0112		
46	0.0022	119	0.0069	229	0.0114	421	0.0157
47	0.0023					427	0.0158
48	0.0024	122	0.0070	235	0.0115	433	0.0159
		124	0.0071	238	0.0116	439	0.0160
50	0.0025	126	0.0072				
51	0.0026	128	0.0073	241	0.0117	446	0.0161
52	0.0027			245	0.0118	453	0.0162
54	0.0028	130	0.0074	249	0.0119	459	0.0163
55	0.0029	132	0.0075				
56	0.0030	134	0.0076	252	0.0120	466	0.0164
58	0.0031	136	0.0077	255	0.0121	470	0.0165
59	0.0032	138	0.0078	258	0.0122		
				263	0.0123		
61	0.0033	140	0.0079	266	0.0124	480	0.0166
63	0.0034	142	0.0080	269	0.0125	487	0.0167
64	0.0035	144	0.0081			493	0.0168
65	0.0036	146	0.0082	273	0.0126		
66	0.0037	148	0.0083	277	0.0127	502	0.0169

Copyright by ASTM Int'l (all rights reserved); Thu Apr 16 07:34:20 EDT 2009
Downloaded/printed by
Laurentian University pursuant to License Agreement. No further reproductions authorized.

TABLE 1 *Continued*

Temperature, °C	Air Correction, %	Temperature, °C	Air Correction, %	Temperature, °C	Air Correction, %	Temperature, °C	Air Correction, %
68	0.0038					510	0.0170
69	0.0039	150	0.0084	280	0.0128	517	0.0171
		154	0.0085	285	0.0129		
70	0.0040	156	0.0086	289	0.0130	521	0.0172
72	0.0041	158	0.0087			534	0.0173
74	0.0042			292	0.0131		
75	0.0043	160	0.0088	296	0.0132	542	0.0174
76	0.0044	163	0.0089			551	0.0175
78	0.0045	165	0.0090	300	0.0133	559	0.0176
		168	0.0091	305	0.0134		
80	0.0046			309	0.0135		
82	0.0047	170	0.0092	313	0.0136	568	0.0177
83	0.0048	173	0.0093	317	0.0137	578	0.0178
587	0.0179	700	0.0190	840	0.0200	1000	0.0209
596	0.0180	712	0.0191	850	0.0201		
606	0.0181	726	0.0192	870	0.0202	1020	0.0210
615	0.0182						
		740	0.0193	880	0.0203	1040	0.0211
620	0.0183	752	0.0194				
635	0.0184			900	0.0204	1060	0.0212
		766	0.0195				
646	0.0185			920	0.0205	1080	0.0213
657	0.0186	780	0.0196				
		790	0.0197	940	0.0206	1100	0.0214
668	0.0187						
679	0.0188	810	0.0198	960	0.0207		
690	0.0189	820	0.0199	980	0.0208		

[A] If the starting temperature, t_0, is above 20°C, the entry in the table opposite that of the starting temperature should be subtracted from each of the succeeding corrections.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

Copyright by ASTM Int'l (all rights reserved); Thu Apr 16 07:34:20 EDT 2009
Downloaded/printed by
Laurentian University pursuant to License Agreement. No further reproductions authorized.