Standard Test Method for
Linear Thermal Expansion of Electrode Carbons[1]

1. Scope*

1.1 This test method covers the determination of the coefficient of linear thermal expansion (CTE) for carbon anodes and cathodes used in the aluminum industry, in baked form, by use of a vitreous silica dilatometer.

1.2 The applicable temperature range for this test method for research purposes is ambient to 1000°C. The recommended maximum use temperature for product evaluation is 500°C.

1.3 This test method and procedure is based on Test Method E228, which is a generic all-encompassing method. Specifics dictated by the nature of electrode carbons and the purposes for which they are used are addressed by this procedure.

1.4 Electrode carbons in the baked form will only exhibit primarily reversible dimensional changes when heated.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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]

E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer

3. Terminology

3.1 *Definitions:*

3.1.1 *linear thermal expansion*, *n*—the change in length per unit length resulting from a temperature change. Linear thermal expansion is symbolically represented by $\Delta L/L_0$, where ΔL is the length change of the specimen ($L_1 - L_0$), L_0 and L_1 are the specimens lengths at reference temperature T_0 and test temperature T_1, respectively. Linear thermal expansion is often expressed as a percentage or in parts per million (such as μm/m).

3.1.1.1 *mean coefficient of linear thermal expansion (CTE), n*—The linear thermal expansion per change in temperature; the mean coefficient of linear thermal expansion is represented by:

$$\bar{\alpha}_{T_1} = \frac{\Delta L/L_0}{\Delta T} = \frac{1}{L_0} \cdot \frac{\Delta L}{\Delta T} = \frac{1}{L_0}\frac{L_1 - L_0}{T_1 - T_0} \tag{1}$$

3.1.1.2 *Discussion*—This has to be accompanied by the values of the two temperatures to be meaningful; the reference temperature (T_0) is 20°C, and the notation may then only contain a single number, such as $\bar{\alpha}_{200}$, meaning the mean coefficient of linear thermal expansion between 20 and 200°C.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *reference specimen*, *n*—a particularly identified or pedigreed material sample, with well-characterized behavior and independently documented performance.

3.2.2 *specimen*, *n*—a representative piece of a larger body (anode, cathode, and so forth) that is considered to be fairly typical of a portion or of the entire piece.

3.2.3 *vitreous silica dilatometer*, *n*—a device used to determine linear thermal expansion, by measuring the difference in linear thermal expansion between a test specimen and the vitreous silica parts of the dilatometer.

4. Summary of Test Method

4.1 A representative specimen is placed into a vitreous silica dilatometer and heated, while its linear expansion is continuously recorded. The change of the specimen length is recorded as a function of temperature. The coefficient of linear thermal expansion is then calculated from these recorded data.

5. Significance and Use

5.1 Coefficients of linear thermal expansion are used for design and quality control purposes and to determine dimensional changes of parts and components (such as carbon anodes, cathodes, and so forth) when subjected to varying temperatures.

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*A Summary of Changes section appears at the end of this standard.

6. Apparatus

6.1 *Dilatometer*—The dilatometer consists of the following:

6.1.1 *Specimen Holder and Push-rod*, both made of vitreous silica. The design of the device shall ensure that the push-rod load on the specimen by itself is not causing deformation. The use of pressure distribution quartz plates on top of the specimen is permissible.

NOTE 1—Dilatometers are usually constructed in horizontal or vertical configurations.[3] Vertical devices are preferred for very large samples and when extensive shrinkage is expected. Horizontal configurations usually afford better temperature uniformity over the specimen, but are subject to drooping when large specimens are employed. Horizontal devices, when used with very large specimens, require special provisions to reduce friction between the specimen and the dilatometer tube to minimize push-rod pressure required to keep the specimen in contact with the end plate. For this application, either configuration is acceptable.

NOTE 2—Multiple rods supporting a platform in place of large diameter tubes have been also used successfully in the vertical configuration.

6.1.2 *Transducer or Indicator*, for measuring the difference in length between the specimen and the dilatometer with an accuracy within $\pm$ 2 μm. The transducer shall translate these movements into an electrical signal suitable for displaying or recording. The non-linearity of this conversion must be less than 0.25 % of the full scale value of the output. The transducer shall be protected or mounted so that the maximum temperature change observed in the transducer during a test will affect the transducer readings by less than 1 μm.

6.1.3 *Temperature Sensors*, for determining the mean temperature of the specimen with an accuracy within $\pm$ 0.5°C. When a thermocouple is used, it shall be referenced (cold junction compensated) to the ice point with an ice-water bath or an equivalent system.

6.1.3.1 Due to the large size of the specimen, a minimum of one thermocouple per 40 mm specimen length must be employed. It is permissible to read the output of each thermocouple independently and average the readings or to connect them in series and divide the single reading by the number of thermocouples to obtain the average. In the latter case, interconnections must be made at or beyond the point of cold junction compensation.

6.1.3.2 The temperature sensors shall be in close proximity to the specimen, preferably in between the quartz dilatometer tube and the specimen. The temperature sensors shall not be directly exposed to the furnace walls.

6.2 *Readout or Recording of Data*:

6.2.1 Manual recording of expansion and temperature values indicated at selected temperature points may be made if the transducer is equipped with or connected to a suitable display and the thermocouples outputs are determined with a potentiometer or millivolt meter.

6.2.2 Chart or data logger recording of the expansion and temperature signals may be accomplished using a device whose resolution is at least 1000 times higher than the expected maximum output signal. All calculations and corrections (see Section 10) must be done externally based on the recorded values.

6.2.3 Computerized recording may be used with similar restriction to 6.2.2. Calculations and corrections may be done using suitable software.

6.3 *Furnace*—The furnace is used for uniformly heating the specimen over the temperature range of interest, but not above 1000°C. Temperature uniformity shall be at least $\pm$ 0.5°C per 50 mm of sample length. The temperatures shall be controlled as a function of time. The furnace may have a muffle (quartz, mullite, alumina, inconel, monel, or stainless steel are most common) or other provisions to provide a protective atmosphere for the specimen. The furnace shall have provisions for continuous purging with an inert gas at a sufficient rate, and exclude air from the specimen while a purge is maintained.

6.4 *Caliper*—The caliper (micrometer or Vernier type) is for measuring the initial length of the specimen, L_0, with an accuracy within $\pm$ 25 μm, and a capacity to open to the length of the specimen plus 1 mm.

7. Test Specimen

7.1 Specimens shall be cylindrical, preferably with a 50 $\pm$ 2 mm diameter. Slightly smaller or larger diameters can also be accommodated without degradation of data. The length of the specimens shall be between 50 and 130 mm in length and have flat and parallel ends to within $\pm$ 25 μm.

7.2 It is permissible to stack up to five disks of smaller lengths to obtain a proper length specimen. The interfaces, however, must be flat and parallel within $\pm$ 25 μm to prevent rocking.

7.3 The dimensions of the specimens should be ordinarily measured as received.

7.4 If water was used in conjunction with their preparation, each specimen must be kept in an oven at 110 $\pm$ 5°C for at least 6 h and allowed to cool down thereafter, prior to testing. If any heat or mechanical treatment is applied to the specimen prior to testing, this treatment should be noted in the report.

8. Calibration

8.1 The transducer should be calibrated by imposing a series of known displacements with a precision screw micrometer, gage blocks, or equally accurate device. For absolute transducers (such as digital encoders, and so forth), this procedure is omitted and periodic verification is sufficient.

8.2 Verification of the calibration of the temperature sensors separately from the dilatometer is to be performed periodically or when contamination of the junction is suspected.

8.3 Regardless of independent calibrations of the transducer and the thermocouples, the dilatometer, as a total system, shall be calibrated by determining the thermal expansion of at least one reference material of known thermal expansion. Recommended reference materials are listed in Annex A1.

8.3.1 The calibration should be done using approximately the same thermal cycle as that used for testing (see 9.7 and 9.8).

8.3.2 The calibration constant may be derived as follows:

$$A = \left(\frac{\Delta L}{L_0}\right)_t - \left(\frac{\Delta L}{L_0}\right)_m \qquad (2)$$

where:

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[3] Hidnert, P. and Krider, H.S., "Thermal Expansion Measurements," *Journal of Research*, National Bureau of Standards, Vol 48, 1952, p. 209.