~~Designation:E2254–08~~ Designation: E2254 – 09

Standard Test Method for Storage Modulus Calibration of Dynamic Mechanical Analyzers[1]

This standard is issued under the fixed designation E2254; 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 describes the calibration or performance confirmation for the storage modulus scale of a commercial or custom built dynamic mechanical analyzer (DMA) over the temperature range of -100 to 300 °C using reference materials in the range of 1 to 200 GPa.

~~1.2SI units are the standard.~~

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

1.3 *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]

E473 Terminology Relating to Thermal Analysis and Rheology

E1142 Terminology Relating to Thermophysical Properties

3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are defined in Terminologies E473 and E1142.

4. Summary of Test Method

4.1 The storage modulus signal ~~measured~~determined by a dynamic mechanical analyzer for an elastic reference material is compared to the reported storage modulus for that reference material. A linear relationship is used to correlate the experimental storage modulus signal with the reported value of the reference material.

4.2 The mode of deformation (for example, tensile, flexure, compression, etc.) shall be reported.

5. Significance and Use

5.1 This test method calibrates or demonstrates conformity of a dynamic mechanical analyzer at an isothermal temperature within the range of -100 to 300 °C.

5.2 Dynamic mechanical analysis experiments often use temperature ramps. This method does not address the effect of that change in temperature on the storage modulus.

5.3 A calibration factor may be required to obtain corrected storage modulus values.

5.4 This method may be used in research and development, specification acceptance, and quality control or assurance.

6. Apparatus

6.1 The essential instrumentation required to provide the minimum dynamic mechanical capability for this test method includes:

6.1.1 *Drive Motor*, to apply force (or displacement) to the specimen in a periodic manner. This motor may also be capable of providing static force or displacement on the specimen.

6.1.2 *Coupling Shaft*, or other means to transmit the force from the motor to the specimen.

6.1.3 *Clamping System(s)*, to fix the specimen between the drive shaft and the stationary clamp(s).

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[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.

Current edition approved ~~Feb.~~Sept. 1, ~~2008.~~2009. Published ~~March 2008.~~January 2010. Original approved in 2003. Last previous edition approved in ~~2003~~2008 as ~~E2254–03.~~E2254–08. DOI: 10.1520/E2254-0~~8~~9.

[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.

6.1.4 *Position Sensor*, to measure the change in position of the specimen during dynamic motion, or,

6.1.5 *Force Sensor*, to measure the force ~~developed by~~applied to the specimen.

6.1.6 *Temperature Sensor*, to provide an indication of the specimen temperature to ± 1 °C.

6.1.7 *Furnace*, to provide controlled heating or cooling of a specimen at a constant temperature or at a constant rate within the applicable temperature range of -100 to +300 °C.

6.1.8 *Temperature Controller*, capable of executing a specific temperature program by operating the furnace between -100 and +300 °C.

6.1.9 *A Data Collection Device*, to provide a means of acquiring, storing and displaying measured or calculated signals, or both. The minimum output signals required are storage modulus, loss modulus, tangent delta, temperature and time.

6.2 Auxiliary instrumentation considered necessary in conducting this method near or below ambient room temperature.

6.2.1 *Cooling* capability to sustain a constant temperature at or below ambient room temperature or to provide controlled cooling.

6.3 *Micrometer*, calipers or other length measuring device capable of measuring length of 1.0 to 100 mm with a precision of ± 0.01 mm.

7. Reagents and Materials

7.1 A reference material of known storage modulus, formed to the shape suitable for characterization by the particular dynamic mechanical analyzer (see Table 1).

NOTE 1—The storage modulus of the calibration materials used in this standard is often similar to that of the construction materials of the test apparatus. Thus the examination of high modulus materials may result in instrument compliance during testing. The test apparatus calibration procedure (see 9.1) should include a compliance correction. The user of this standard shall verify whether or not such compliance corrections are included prior to its use.

8. Sampling

8.1 Test specimens are typically prepared in the form of a rectangular test bars or film strips.

NOTE 2—It is common practice to bevel or "break" edges of machined parts. This practice shall not be followed in the preparation of test specimens for this method. The measured storage modulus of such test specimens reads low due to imperfect sample geometry.

9. Calibration and Standardization

9.1 Perform any storage modulus signal calibration procedures recommended by the manufacturer of the dynamic mechanical analyzer as described in the operations manual.

10. Procedure

10.1 Prepare the dynamic mechanical analyzer for operation under the test conditions (for example, specimen clamps, purge gas, etc.) to be used for the characterization of the test specimens. Unless otherwise indicated, the temperature condition shall be isothermal between 20 and 22 °C.

10.2 Ensure that the storage modulus signal is less than 1 MPa with no test specimen loaded and at an oscillation test frequency of 1 Hz.

NOTE 3—Alternatively, a thin specimen of a low modulus material (for example, a thin piece of paper) may be used. The dimensions of the test specimen (see 10.3) shall be used rather than the true dimensions of the thin low modulus material.

TABLE 1 Reference Material Modulus[A,B,C]

Storage Modulus, GPa					
Temperature, °C	Carbon Steel[D]	Monel[E]	Copper[F]	Aluminum	UHMWPE
-198	207	185	121	77.9	. . .
-101	201	182	116	75.8	. . .
-46	198	180	114	74.5	. . .
21	192	179	114	73.1	1.26
93	191	179	112	71.7	. . .
149	189	178	112	70.3	. . .
204	186	177	110	65.5	. . .
260	182	175	. . .	. . .	. . .
316	177	170	. . .	. . .	. . .

[A] American Society of Mechanical Engineers, New York, NY, B31.5a, *Refrigeration Piping*, page 45 (1992).

[B] *Perry's Chemical Engineers' Handbook*, R.H. Perry, D.W. Green, J.O. Maloney (eds.), 6th Edition, McGraw-Hill, New York, NY, page 6-92 (1984).

[C] *Ultra High Molecular Weight Polyethylene Standard Reference Material SRM 8456*, National Institute of Standards and Technology, Gaithersburg, MD 20899 (2000).

[D] 3.5 % Ni, <0.30 C.

[E] 67 % Ni, 30 % Cu.

[F] 99.90 % Cu, Alloy C12000, C12200.