Designation: C1735 – 17

Standard Test Method for Measuring the Time Dependent Modulus of Sealants Using Stress Relaxation[1]

This standard is issued under the fixed designation C1735; 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 a procedure for measuring the time dependence of modulus in elastomeric joint sealants in a test specimen configuration described in Test Method C719. These sealant materials are typified by highly filled rubber materials. Any Mullins effect is first assessed and mitigated in two loading-unloading cycles. Time dependence of modulus in materials is then determined using a stress relaxation procedure.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

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]

C717 Terminology of Building Seals and Sealants
C719 Test Method for Adhesion and Cohesion of Elastomeric Joint Sealants Under Cyclic Movement (Hockman Cycle)
E4 Practices for Force Verification of Testing Machines
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E631 Terminology of Building Constructions
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminologies E631 and C717.

4. Summary of Test Method

4.1 This test method consists of two loading cycles where strain is increased from a 0 % to a user-defined maximum strain and one stress relaxation procedure at a strain no greater than ⅔ of the user-defined maximum strain. A schematic diagram of this test strain history is shown in Fig. 1.

4.2 The motivation for the two loading-unloading cycles is to assess if any Mullins effect is present and to mitigate any of this effect in the subsequent stress relaxation test. As long as the maximum strain achieved during the first deformation is not exceeded, however, all subsequent loadings follow the same stress-strain curve.

4.3 Stress relaxation procedure is based on a sudden imposition of either a tensile or compressive strain on the test specimen at a value of no more than ⅔ of the maximum strain and the measurement of the load required to maintain this tensile or compressive strain as a function of time.

4.4 The conversion of load relaxation values to apparent modulus and fractional change in apparent modulus is accomplished by inputting information about the specimen geometry and the amount of deformation into an equation that is based on the statistical theory of rubber-like elasticity.

5. Significance and Use

5.1 The intent of this test method is to determine the time dependence of modulus in building joint sealants using two loading-unloading cycles to identify and mitigate any Mullins effect, and followed by a stress relaxation procedure to determine the time dependent modulus.

5.2 This test method has found applications in screening the performance of building joint sealants since the modulus is one indicator of the ability of elastomeric building sealant to withstand environmental induced movements.

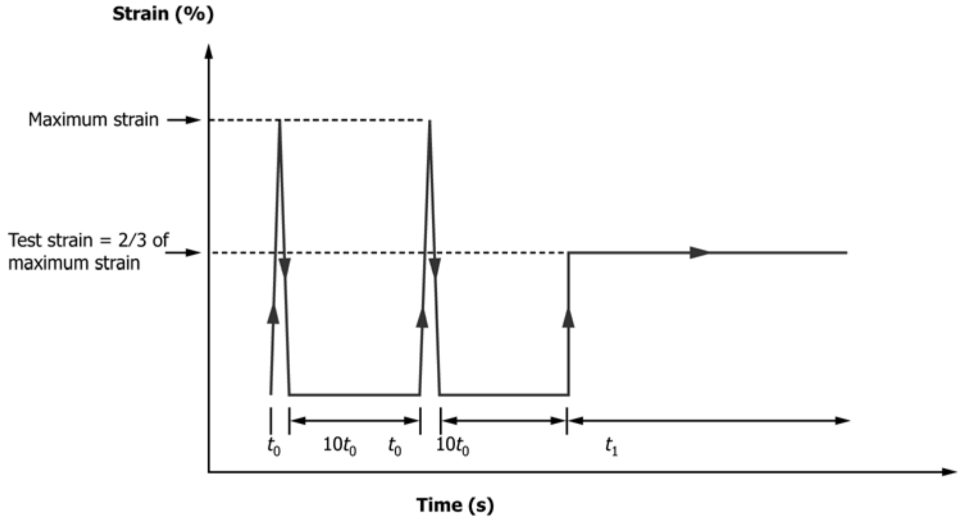
6. Apparatus

6.1 *Testing Machine*—Any testing machine in compliance with Practices E4, capable of producing a constant crosshead

[1] This test method is under the jurisdiction of ASTM Committee C24 on Building Seals and Sealants and is the direct responsibility of Subcommittee C24.20 on General Test Methods.

Current edition approved Jan. 1, 2017. Published February 2017. Originally approved in 2011 as C1735-11. Last previous edition C1735-11. DOI: 10.1520/C1735-17.

[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.



The value of t_0 is 20 s and t_1 is typically 2000 s.

FIG. 1 Strain History Used for Two Loading-Unloading Cycles and a Stress Relaxation Measurement

displacement rate in displacement control and equipped with means for recording complete load versus displacement curves during the test.

6.2 *Load Cell*—The load cell of the testing machine shall be capable of recording the load with an accuracy of ±1 % of the maximum indicated value.

6.3 *Loading Fixtures*—Used to mount specimens to the testing machine so that the surface of the substrate is perpendicular to the direction of the applied load and to minimize any eccentric loading in the test specimen.

6.4 *Air-circulating Oven*—To condition specimens at the specific temperature and relative humidity.

6.5 *Mechanical Fastener or Rubber Bands*—To hold a specimen assembly together before and after filling it with sealant compound.

6.6 *Geometry Measuring Tool*—To measure the dimensions of aluminum substrate and sealant to an accuracy of at least ±5 mm (0.2 in.). Because stress is the load per area exerted on the test specimen, an accurate measurement of the geometry is critical.

7. Preparation of Test Specimen

7.1 Test conditions of temperature and relative humidity used throughout this test method are defined in Terminology C717.

7.2 The standard substrate used in the test shall be aluminum alloy (76.2 by 12.7 by 12.7 mm) (3 by 0.5 in. by 0.5 in.), 6063-T5, or 6061-T6 with anodizing process AA-M10C22A31. Prior to use, the aluminum alloy shall be cleaned according to a procedure described in the Test Method C719, which involves cleaning the substrate by wiping the surface with methyl ethyl ketone or similar solvent. Then dip the surface in a detergent solution. An alternative would be a 0.1 % solution of a clear hand dishwashing detergent. These solutions should be made up in distilled or deionized water. Rinse the surface (without touching it) in distilled or deionized water and allow it to air dry.

7.3 Where use of primer is recommended by the sealant manufacturer, substrate materials shall be primed with the recommended primer or primers.

7.4 Mix thoroughly for 5 min at least 250 g of base compound with the appropriate amount of curing agent being careful not to generate excess heat. For single-component sealants, no mixing of components is required. Dispense the compound from a cartridge into a specimen cavity (50.8 by 12.7 by 12.7 mm) (2 by 0.5 by 0.5 in.) formed by two parallel substrate faces (50.8 by 12.7 mm) (2 by 0.5 in.) with a polytetrafluoroethylene (PTFE) film on the back and PTFE spacers (12.7 by 12.7 by 12.7 mm) (0.5 by 0.5 by 0.5 in.) on each end, as shown in Fig. 2.

7.5 Use appropriate mechanical fasteners or rubber bands to hold the specimen cavity together before and after filling it with the compound.

7.6 Condition the specimen in the fixture at the standard conditions of temperature and relative humidity for a minimum of 5 h. Then remove the mechanical fasteners or rubber bands and the PTFE backing film. Keep the PTFE spacers and the aluminum substrate intact.

7.7 Allow specimen to cure according to the Test Method C719 specifications.

7.8 Test specimens surfaces should be smooth and free from nicks and scratches.

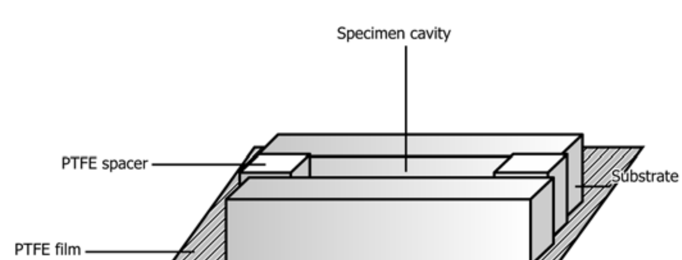


FIG. 2 Illustration of Specimen Preparation Before Filling it with the Sealant Compound

7.9 The finished test specimen is shown in Fig. 3.

8. Number of Test Specimens

8.1 Test at least three specimens of each sealant.

9. Procedure

9.1 Perform the test at the standard conditions of temperature and relative humidity stated in 7.1. Tests at other ambient temperature and relative humidity may be run if desired.

9.2 Mount the specimen (as prepared in Section 7) in the fixture of the testing machine. Exercise precautions to minimize axial misalignment.

9.3 Set the testing machine at a crosshead displacement rate of 20 mm/min (0.787 in./min). Set the direction of applied strain, either tensile or compressive.

9.4 Apply load to the specimen until the strain reaches the maximum strain, after which the specimen is completely unloaded at the same crosshead displacement rate.

9.5 The specimen is allowed to rest for 200 s. This is done to ensure the viscoelastic recovery from one loading is complete before the next loading is initiated.

9.6 Repeat 9.4 and 9.5 for the second loading-unloading cycle.

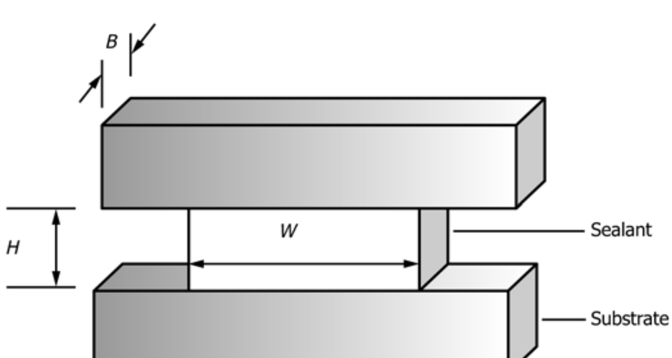
9.7 Set the testing machine at a crosshead displacement rate of 1000 mm/min (39.37 in./min).

9.8 Apply load to the specimen until the strain reaches the desired test strain, no greater than ⅔ of the maximum strain and hold that value while load is monitored as a function of time. The time required to load the specimen is less than 1 s so the first data point is not taken until after 10 s to avoid transient effects associated with loading.

9.9 Inspect the sample to note the locus of joint failure, if any failure occurs.

10. Calculation and Analysis

10.1 Calculate an apparent modulus, E_a, using a relationship based on the statistical theory of rubber-like elasticity:



H, *B* and *W* are the width, depth and length of the specimen, respectively.

FIG. 3 Geometry for the Sealant Specimen

$$Ea\left(t,\lambda\right) = \frac{3L(t)}{WB\left(\lambda - \lambda^{-2}\right)} \quad (1)$$

where:

L = load, N (lb),
W = length of the specimen, m (in.),
B = depth of the specimen, m (in.),
t = time, s,
λ = extension ratio, which is given by:

$$\lambda = 1 + \frac{\Delta}{H} \quad (2)$$

where:

Δ = crosshead displacement of testing machine, and
H = width of the specimen, m (in.).

10.2 For convenience in comparing the relative change in apparent modulus of materials during environmental exposures, the fractional change in apparent modulus, *F*, may be calculated as a function of time:

$$F = \frac{E_a(t_1)}{E_a(t_0)} \quad (3)$$

where:

$E_a(t_0)$ = apparent modulus at time, t_0, and
$E_a(t_1)$ = apparent modulus at time, t_1.

10.3 A plot of the fractional change in apparent modulus is useful for comparison between different exposure times. For such a graph, no change would be represented as a horizontal straight line at $F = 0$. A horizontal line above or below $F = 0$ indicates that exposure causes a vertical shift in the stress relaxation curve but no change in shape (that is, the time dependence does not change. Something other than a horizontal straight line indicates a change in time dependence.

11. Report

11.1 Report the following information:
11.1.1 Identification of the sealant tested, including type, source, manufacturer code number, curing conditions employed,
11.1.2 Identification of the substrate used,
11.1.3 Name and description of primers used, if any,
11.1.4 Temperature and relative humidity,
11.1.5 Number of specimens tested,
11.1.6 Type of strain (compressive or tensile),
11.1.7 Maximum strain, test strain.
11.1.8 Descriptive of the type of failure, if any:
11.1.8.1 Cohesive failure, if separation occurred within the material,
11.1.8.2 Adhesive failure, if separation occurred at the interface of the substrate and the sealant,
11.1.8.3 Mixed failure, if both adhesive and cohesive failure regions are present.
11.1.9 Records in terms of apparent modulus or normalized modulus as a function of time.

12. Precision and Bias

12.1 The precision of this test method is based on an inter-laboratory study of C1735 Standard Test Method for Measuring the Time Dependent Modulus of Sealants Using

Stress Relaxation, conducted in 2016. Seven laboratories tested a single type of sealant. Every "test result" represents an individual determination, and each laboratory reported triplicate test results. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. C24-1063.[3]

12.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

12.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

12.1.1.2 Repeatability limits are listed in Table 1.

12.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

12.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

12.1.2.2 Reproducibility limits are listed in Table 1.

12.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

12.1.4 Any judgment in accordance with 12.1.1 and 12.1.2 would have an approximate 95 % probability of being correct.

TABLE 1 Modulus

Material	Average, $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	Repeatability Limit r	Reproducibility Limit R
Silicone Sealant	137.38	6.76	11.33	18.93	31.72

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

12.3 The precision statement was determined through statistical examination of eighteen test results, as reported by six of the seven participating laboratories, on a single type of sealant material. The sealant tested was described as a commercial silicone sealant formulation prepared according to Test Method C719.

13. Keywords

13.1 elastomeric joint sealant; environmental attack; Hockman cycle; modulus; Mullins effect; stress relaxation

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:C24-1063.