



Designation: E3098 – 24

Standard Test Method for Uniaxial Pre-strain and Thermal Free Recovery of Shape Memory Alloys¹

This standard is issued under the fixed designation E3098; 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 thermal cycling of a Shape Memory Alloy (SMA) specimen through the phase transformation under minimal load transformation after uniaxial deformation. This method will measure residual strain after loading and unloading, recovered strain on heating, total unrecovered strain upon cooling, and transformation temperatures. This test is done to provide data for the selection of SMA materials, quality control, design allowables and actuator design.

1.2 *Units*—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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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

- E4 Practices for Force Calibration and Verification of Testing Machines
- E6 Terminology Relating to Methods of Mechanical Testing
- E8/E8M Test Methods for Tension Testing of Metallic Materials

- E9 Test Methods of Compression Testing of Metallic Materials at Room Temperature
- E21 Test Methods for Elevated Temperature Tension Tests of Metallic Materials
- E83 Practice for Verification and Classification of Extensometer Systems
- E209 Practice for Compression Tests of Metallic Materials at Elevated Temperatures with Conventional or Rapid Heating Rates and Strain Rates
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E1169 Practice for Conducting Ruggedness Tests
- E2368 Practice for Strain Controlled Thermomechanical Fatigue Testing
- E3097 Test Method for Uniaxial Constant Force Thermal Cycling of Shape Memory Alloys
- F2004 Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis
- F2005 Terminology for Nickel-Titanium Shape Memory Alloys
- F2082 Test Method for Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery
- F2516 Test Method for Tension Testing of Nickel-Titanium Superelastic Materials

2.2 Other Standards:

- IEEE/ASTM SI 10 American National Standard for Metric Practice²
- ASQ C1 General Requirements for a Quality Program³
- ISO 9001 Quality Management Systems—Requirements⁴

3. Terminology

3.1 *Definitions*—The terms specific to this test method are defined in this section. All other terms used in this test method are in accordance with Terminologies in F2005, and E3097. Transformation temperatures and transformation strains are determined in accordance with E3097 as applied to the E3098

¹ This test method is under the jurisdiction of ASTM Committee E08 on Fatigue and Fracture and is the direct responsibility of Subcommittee E08.05 on Cyclic Deformation and Fatigue Crack Formation.

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

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

strain-temperature response shown in Fig. 1 and Fig. 2, respectively, unless otherwise stated.

3.2 *recovery strain* (e_{rec})—The amount of residual strain that is recovered in the specimen after heating to the UCT and cooling to the LCT following pre-straining, it is equal to the unloaded strain (e_u) minus strain at lower cycle temperature (e_{LCT}) after cooling from the UCT.

3.3 *stress at maximum strain* (S_i)—The stress in the specimen at the maximum loading strain (e_i).

3.4 *two way strain* (e_{TW})—The cooling transformation strain generated by cooling through the phase transformation with an applied tensile stress of 7 MPa or less. $e_{TW} = e_{ct}$ (under applied stress < 7 MPa).

3.5 *unloaded strain* (e_u)—The specimen strain at LCT after pre-straining and then unloading, but prior to heating.

3.6 Abbreviations:

3.6.1 *LCT*—Lower Cycle Temperature

3.6.2 *UCFTC*—Uniaxial Constant Force Thermal Cycling

3.6.3 *UCT*—Upper Cycle Temperature

3.6.4 *UPFR*—Uniaxial Pre-strain and Thermal Free Recovery

3.6.5 *TWSME*—Two-way Shape Memory Effect

3.7 See also E6: General Terminology

4. Summary of Test Method

4.1 This test method involves cooling a test specimen to its fully martensitic state, deforming the specimen to a defined strain under uniaxial loading, removing the force, heating the specimen to its fully austenite phase, and then cooling the specimen to its fully martensite phase. During heating and cooling the extension or contraction of the specimen is measured and plotted versus the specimen temperature.

5. Significance and Use

5.1 This test method is used to measure a specimen's material and shape memory effect properties in response to a uniaxial deformation and then cycling through a full thermal transformation to recover all or a portion of the deformation. A material's martensitic stress-strain response, martensite re-

sidual strain, austenite recovered strain, and unrecovered strain (or plastic deformation) after thermal cycling is determined.

5.2 Measurement of the specimen's motion closely parallels many shape memory applications and provides a result that is applicable to the function of the material.

5.3 This test method may be used for, but is not limited to, wire, round tube, or strip samples. It is able to provide an assessment of the product in its semi-finished form.

5.4 This test method provides a simple method for determining transformation temperatures by heating and cooling specimens through their full thermal transformation after uniaxial pre-straining in the martensite state.

5.5 This test method can be used on trained and processed material in a semi-finished form to measure Two Way Shape Memory Effect (TWSME) by comparing the strain in the austenite state and martensite states with minimal applied stress. The force is set to a minimum value not to exceed a corresponding stress of 7 MPa (in accordance with Test Method F2516).

5.6 This test method is useful for quality control, specification acceptance, and research.

5.7 Transformation temperatures derived from this test method may not agree with those obtained by other test methods due to the effects of strain and stress on the transformation.

5.8 Components such as springs or other semi-finished parts can be tested using this method as agreed upon by the customer and supplier. Units of stress and strain can be replaced with force and displacement.

6. Interferences

6.1 The initial condition of the test specimen can significantly impact test results.

NOTE 1—Care should be taken to assure the material is free of unintended residual stresses from fabrication, processing, or handling. Cutting and grinding can cause cold work which affects the transformation temperatures. Oxidation during heat treatment can change the thermal properties of the specimen and affect the temperature uniformity. Such effects are magnified by specimens with smaller gauge diameters.

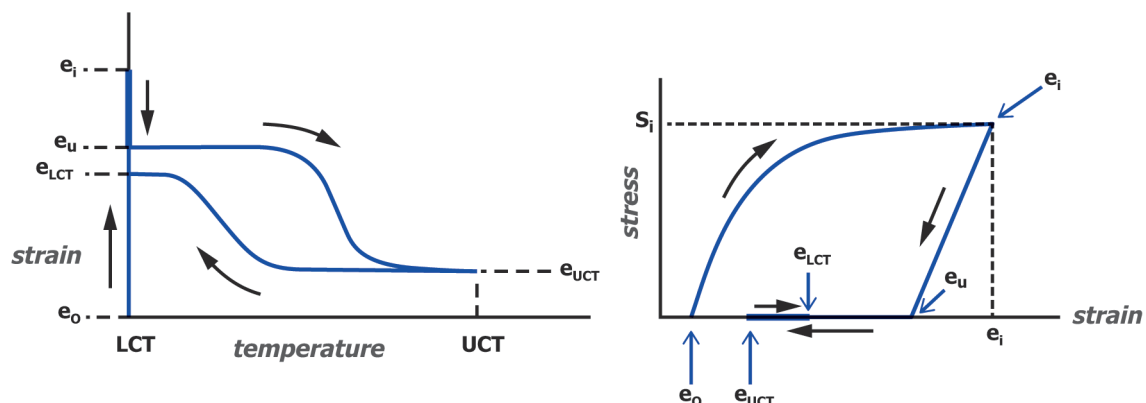


FIG. 1 Strain-Temperature and Stress-Strain Diagrams

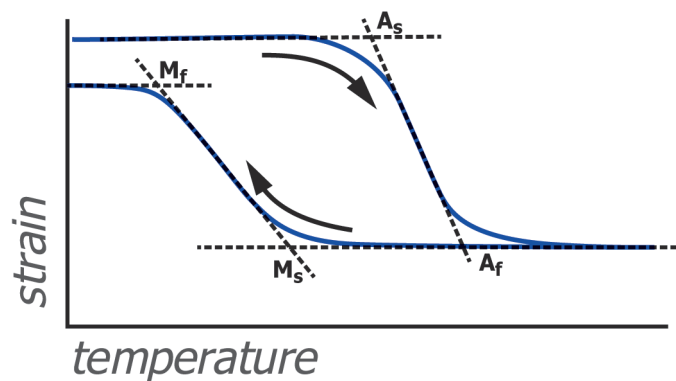


FIG. 2 Transformation Temperatures by Intersection of Tangent Lines

6.2 When testing wire, make sure that the gripping mechanism does not cause errors in strain measurement, for example slipping in the grips.

6.3 For tension and compression testing the extensometer design and size shall be chosen so that the extensometer measures all deformation within the reduced gauge length of the sample.

6.4 Complete thermal transformation is required for accurate results. The material's martensite finish and austenite finish temperatures may be estimated prior to the test by Differential Scanning Calorimetry (Test Method F2004), or Bend and Free Recovery (Test Method F2082).

6.5 Make sure that the heating and cooling system maintains a uniform specimen temperature within $\pm 3^\circ\text{C}$, along the specimen length, over the gauge section. Temperature gradients in the specimen will affect the apparent transformation temperatures and strains. Please see 10.1 for details on temperature measurement.

6.6 The heating and cooling rate for the test shall be consistent with the sample thickness so that the test section of the specimen is at a uniform temperature within $\pm 3^\circ\text{C}$, transverse to the specimen length, over the gauge section. Please see 10.1 for additional details on temperature measurement.

NOTE 2—Interferences 6.5 and 6.6 may be achieved by selecting hold times at the UCT and LCT to insure the specimen and temperature control system are fully equilibrated before starting/continuing the thermal cycle.

6.7 Make sure the specimen is fully austenitic at the upper cycle temperature (UCT) for all stress levels to be tested. This is shown graphically in Test Method E3097, Figure 2. It is selected to be higher than the A_f determined by a DSC test per Test Method F2004. For example, a temperature between 10°C to 100°C above A_f may be selected in consideration of the stress applied to the specimen. The DSC test shall be done on the sample material from the same lot and with the same thermomechanical history as the UPFR test material.

6.8 Make sure that the specimen is fully martensitic at the lower cycle temperature (LCT). It is selected to be 10°C to 30°C lower than M_f determined by a DSC test per Test Method F2004. However, the DSC test shall be done on the sample material from the same lot and with the same thermomechanical history as the UPFR test material.

NOTE 3—Transformation temperatures will vary with applied stress to the specimen and also vary from alloy to alloy subjected to the same stress. For unfamiliar alloys it is recommended that a range of stresses be tested to assess the effect of stress on transformation temperatures prior to any extensive test program.

6.9 The output signal of a mechanical extensometer may change as a function of temperature. See Practice E83, Appendix X2. A thermal compensation routine shall be developed to compensate for the changes in the output signal. See 9.2.

6.10 Transformation temperatures will vary with the pre-strain applied to the specimen and also vary from alloy to alloy subjected to the same strain. For unfamiliar alloys it is recommended that a range of pre-strains be tested to assess the effect of the maximum strain prior to any extensive test program. For example, plateau strain under uniaxial loading can be used to establish initial test conditions (Test Methods F2516).

6.11 In the absence of a specified pre-strain limit, the specimen pre-strain can be a minimum of the 1% yield offset as defined by the offset method described in Test Method E8/E8M.

7. Apparatus

7.1 The tension apparatus is as described in Test Methods E8/E8M.

7.2 The compression testing machine bearing blocks and strain transducer shall be as described in Test Methods E21 or Practice E209.

7.3 The heating and cooling apparatus and the temperature measuring apparatus shall be as described in Test Methods E21 for high temperature tension testing and Practice E209 for compression testing.

7.4 The test apparatus shall be capable of controlling test specimen temperature in air between the LCT and UCT with a temperature uniformity of $\pm 3^\circ\text{C}$ in the axial and transverse direction over the gauge section. See 10.1 for details on temperature measurement.

8. Sampling, Test Specimens, and Test Units

8.1 The number and location of samples from each lot of material shall be agreed upon between the customer and the supplier.