Designation: E3098 – 17

Standard Test Method for Mechanical Uniaxial Pre-strain and Thermal Free Recovery of Shape Memory Alloys[1]

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 heating and cooling a Shape Memory Alloy (SMA) specimen through transformation after uniaxial deformation to determine residual strain after loading and unloading, recovered strain on heating, total unrecovered strain upon cooling, and transformation temperatures.

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 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
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E74 Practice of Calibration of Force-Measuring Instruments for Verifying the Force Indication of Testing Machines
E83 Practice for Verification and Classification of Extensometer Systems
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
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
F2004 Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis
F2005 Terminology for Nickel-Titanium Shape Memory Alloys
F2063 Specification for Wrought Nickel-Titanium Shape Memory Alloys for Medical Devices and Surgical Implants
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 Practice[2]
ASQ C1 General Requirements for a Quality Program[3]
ISO 9001 Quality Management Systems—Requirements[4]

3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are found in Terminology F2005:

[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 service@astm.org. 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.

3.2 *austenite finish strain (e_{Af})*—Strain at the austenite finish temperature.

3.3 *austenite start strain (e_{As})*—Strain at the austenite start temperature.

3.4 *initial Strain (e_0)*—Specimen strain at LCT after normalizing (see 11.1) and prior to pre-straining the specimen.

3.5 *lower cycle temperature (LCT)*—LCT is the minimum temperature of the thermal cycle. It is selected to be 10 to 30°C lower than M_f determined by a DSC test in accordance with Test Method F2004. However, the DSC test shall be done on the sample material in the same condition as the UPFR test material.

3.6 *maximum loading strain (e_i)*—Maximum specimen strain during pre-straining at the LCT.

3.7 *recovery strain (e_{rec})*—Is 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.

3.8 *strain at lower cycle temperature (e_{LCT})*—Specimen strain at the LCT after pre-straining and unloading at the LCT and heating to the UCT and cooling to LCT.

3.9 *strain at upper cycle temperature (e_{UCT})*—Specimen strain at the UCT after pre-straining and unloading at the LCT and heating to the UCT.

3.10 *stress (S)*—Stress is defined as the ratio of force to the specimen original cross sectional area.

3.11 *transformation strain (e_t)*—The strain recovery due to the austenitic transformation obtained when heating at a specified stress. $e_T = e_{As} - e_{Af}$

3.12 *two way strain (e_{TW})*—Specimen strain at the LCT after cooling from the UCT minus the strain at the UCT. This is the strain induced in the shape memory alloy specimen when it is cooled from UCT to LCT with an applied tensile stress of 7 MPa or less. $e_{TW} = e_{LCT} - e_{UCT}$

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

3.14 *upper cycle temperature (UCT)*—UCT is the maximum temperature of the thermal cycle. It is selected to be higher than the A_f determined by a DSC test in accordance with Test Method F2004. For example, a temperature between 10 to 100 °C above A_f may be selected in consideration of the strain applied to the specimen. The DSC test shall be done on the sample material in the same condition as the UPFR test material.

3.15 *Abbreviations:*

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

3.16 See also E4: 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 austenitic 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 martensite stiffness, martensite residual 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 by comparing the strain in the austenite state and martensite states with no applied stress.

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.

6.2 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).

6.3 Make sure that the heating and cooling system maintains a uniform specimen temperature within ± 3ºC, 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.

6.4 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 ± 3ºC, transverse to the specimen length, over the gauge section. See 10.1 for additional details on temperature measurement.

NOTE 2—Interferences 6.3 and 6.4 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.

6.5 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 Method F2516).

6.6 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 Methods E8/E8M.

6.7 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.

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 E9 or Practice E209.

7.3 The heating and cooling apparatus and the temperature measuring apparatus shall be as described in Test Method 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 a minimum temperature of M_f -30°C and a maximum temperature of A_f +100°C with a temperature uniformity of ± 3°C in the axial and transverse direction overt 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.

8.2 Tensile specimens are as described in Test Methods E8/E8M for different product forms except that the gage length needs to be a minimum of one (1) times the diameter.

8.3 For wires, strain may be measured from displacement between the grips.

8.4 Compression specimens are described in Test Methods E9 and Practice E209.

9. Calibration and Standardization

9.1 The tension or compression testing system shall be calibrated and verified according to Practices E4.

9.2 An extensometer system shall be verified according to Practice E83 Class B-2. The strain signal of a mechanical extensometer will change as a function of temperature. See Practice E83, Appendix X2. To compensate for a thermally induced shift in an extensometer a zero-force thermal strain compensation routine shall be used. The extensometer is attached to a specimen having a known coefficient of thermal expansion. For example, NIST Standard Reference Material 731L1, borosilicate glass, has been shown to be a suitable material. Using the same thermal cycle profile as the planned test, including the UCT, LCT, and the heating and cooling rates, the extensometer output signal is recorded over a complete thermal cycle. The thermal effect on the offset is determined by the difference between the extensometer output and the known thermal expansion. The determined thermal response shall then be used within the test's thermal cycle to provide strain compensation values.

10. Heating and Cooling

10.1 Measurement and control of temperature, temperature gradients and heating/cooling rates shall be employed in accordance with Practice E2368, Section 7.4. In this instance, T_{max} = UCT and due to the hysteretic behavior of SMA, Practice E2368, Section 7.4.5 should be disregarded. The maximum allowable gradient shall be ±3°C in both the axial and transverse direction, over the gauge section.

11. Procedure

11.1 *Normalizing:*

11.1.1 The specimen shall be mounted in a tensile/compression load system at room temperature with the entire test system equilibrated at room temperature. The load is set to a minimum force not to exceed 7 MPa (in accordance with Test Method F2516).

11.1.2 The specimen is heated to the UCT, cooled to the LCT and held at the temperature and stress for a time sufficient to equilibrate the temperature and strain. The heating and cooling rates for normalizing are not restricted to a specific rate but shall be sufficient to ensure temperature equilibration at the UCT and LCT, as specified in 10.1.

11.2 Adjust the specimen temperature to M_f -10ºC or less, or the temperature specified in the test plan or product specification.

11.3 Strain the specimen to the strain level specified in the test plan or product specification. Suitable limits for speed of testing shall be specified for materials for which the differences resulting from the use of different speeds are of such magnitude that the test results are unsatisfactory. In the absence of a specified rate, a strain rate of 0.001mm/mm to 0.01 mm/mm per minute shall be used.

11.4 Unload the specimen to the stress of 7 MPa as specified in 11.1. Hold the temperature and stress for a time sufficient to equilibrate the temperature and strain.