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Standard Test Method for Uniaxial Constant Force Thermal Cycling of Shape Memory Alloys[1]

This standard is issued under the fixed designation E3097; 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 defines procedures for thermomechanical cycling of shape memory alloy (SMA) material and components under constant force. This method characterizes the transformation properties such as transformation temperatures, actuation strain and residual strain, when a SMA is thermally cycled through the phase transformation under a constant applied force. 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
F2004 Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis
F2005 Terminology for Nickel-Titanium Shape Memory Alloys
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 Specific technical terms used in this test method are found in Terminology F2005.

3.2 *Definitions:*

3.2.1 *actuation strain (e_{act})*—The full strain recovery obtained when heating from LCT to UCT at a specified stress. It includes the thermal expansions of martensite and austenite as well the phase transformation strain. $e_{act} = e_{LCT} - e_{UCT}$

3.2.2 *austenite 50 % (A_{50})*—Temperature at which the transformation from martensite to austenite is 50 % completed.

[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.

$A_{50} = (A_s + A_f)/2$.

3.2.3 *austenite finish strain* (e_{Af})—Strain calculated at the austenite finish temperature during final heating.

3.2.4 *austenite start strain* (e_{As})—Strain calculated at the austenite start temperature during final heating.

3.2.5 *cooling transformation strain* (e_{ct})—The strain recovery due to the martensitic transformation obtained when cooling at a specified stress. $e_{ct} = e_{Ms} - e_{Mf}$

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

3.2.7 *hysteresis width (HWIDTH)*—Width of the thermal hysteresis curve in degrees centigrade. Distance on the temperature axis between a vertical line drawn through the A_{50} point and a vertical line drawn through the M_{50} point.

3.2.8 *initial loading strain* (e_i)—Initial specimen strain after normalization and before cooling when loaded at the UCT.

3.2.9 *initial strain* (e_0)—Specimen strain at UCT after normalizing (see 11.1) and prior to loading the specimen.

3.2.10 *lower cycle temperature (LCT)*—Minimum temperature of the thermal cycle.

3.2.11 *martensite 50 % (M_{50})*—Temperature at which the transformation from austenite to martensite is 50 % completed. $M_{50} = (M_s + M_f)/2$.

3.2.12 *martensite finish strain* (e_{Mf})—Strain calculated at the martensite finish temperature during cooling. (See Figure 1)

3.2.13 *martensite start strain* (e_{Ms})—Strain calculated at the martensite start temperature during cooling.

3.2.14 *residual strain* (e_{res})—The final strain at the upper cycle temperature minus the initial strain at the upper cycle temperature. $e_{res} = e_{UCT} - e_i$

3.2.15 *strain at the lower cycle temperature* (e_{LCT})—Specimen strain at the LCT after cooling from the UCT to the LCT under the specified stress. (See Fig. 1.)

3.2.16 *strain at the upper cycle temperature* (e_{UCT})—Specimen strain at the UCT after cooling to the LCT and heating to the UCT at the specified stress. (See Fig. 1.)

3.2.17 *thermal transformation span (TSPAN)*—Thermal transformation span in degrees centigrade at a specified stress. Distance on the temperature axis between a vertical line drawn through the A_f point and a vertical line drawn through the M_f point. $TSPAN = A_f - M_f$.

3.2.18 *upper cycle temperature (UCT)*—The maximum temperature of the thermal cycle.

3.3 *Abbreviations:*

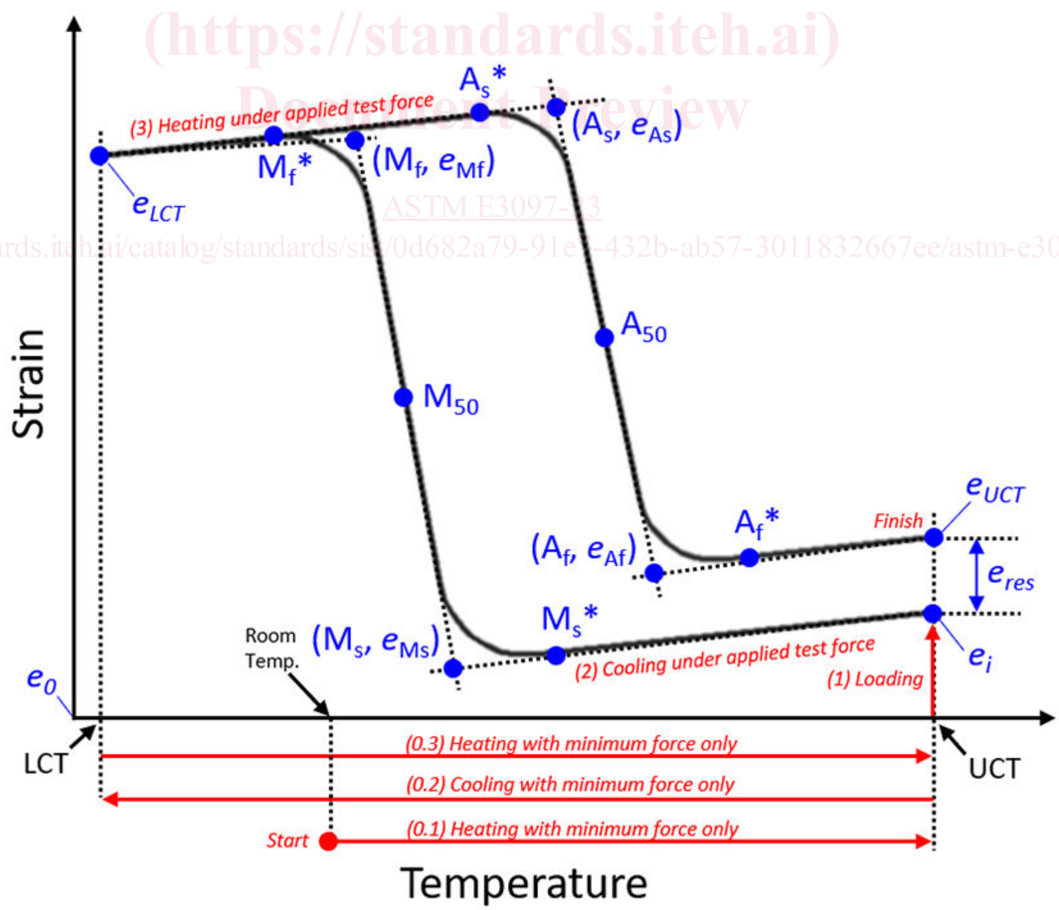

FIG. 1 Typical Constant Force Thermal Cycle and Test Methods Terms

3.3.1 *UCFTC*—Uniaxial Constant Force Thermal Cycling

3.4 See also Terminology E6.

4. Summary of Test Method

4.1 Using a conventional uniaxial tension or compression testing apparatus (or a dead weight loading system) with a temperature control chamber (or other system for heating and cooling at a controlled rate) the material is heated to the UCT, above the austenite finish (A_f) temperature, loaded to a specified stress, then cooled to the LCT, a temperature below the martensite finish (M_f) temperature, and then heated to the UCT.

5. Significance and Use

5.1 Constant force thermal cycling tests determine the effect of stress on the transformation temperatures, recovered strain and residual strain of a shape memory alloy. The tests may be for one thermal cycle. A standard test method for force controlled repeated thermal cycling of shape memory alloys is currently under development.

5.2 Measurement of the specimen's thermomechanical behavior 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. Thus 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 under force.

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 minimal applied stress. The force is set to a minimum value not to exceed a corresponding stress of 7 MPa (in accordance 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.

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).

6.5 Make sure that the heating and cooling system maintains a uniform specimen temperature within ± 3 °C, along the specimen length, over the gauge section. Temperature gradients in the specimen will affect the apparent transformation temperatures and strains. 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 ± 3 °C, transverse to the specimen length, over the gauge section. See 10.1 for details on temperature measurement.

NOTE 2—Requirements specified in interferences 6.5 and 6.6 may be achieved by selecting hold times at the UCT and LCT to ensure 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 Fig. 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 UCFTC 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 UCFTC 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.

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.

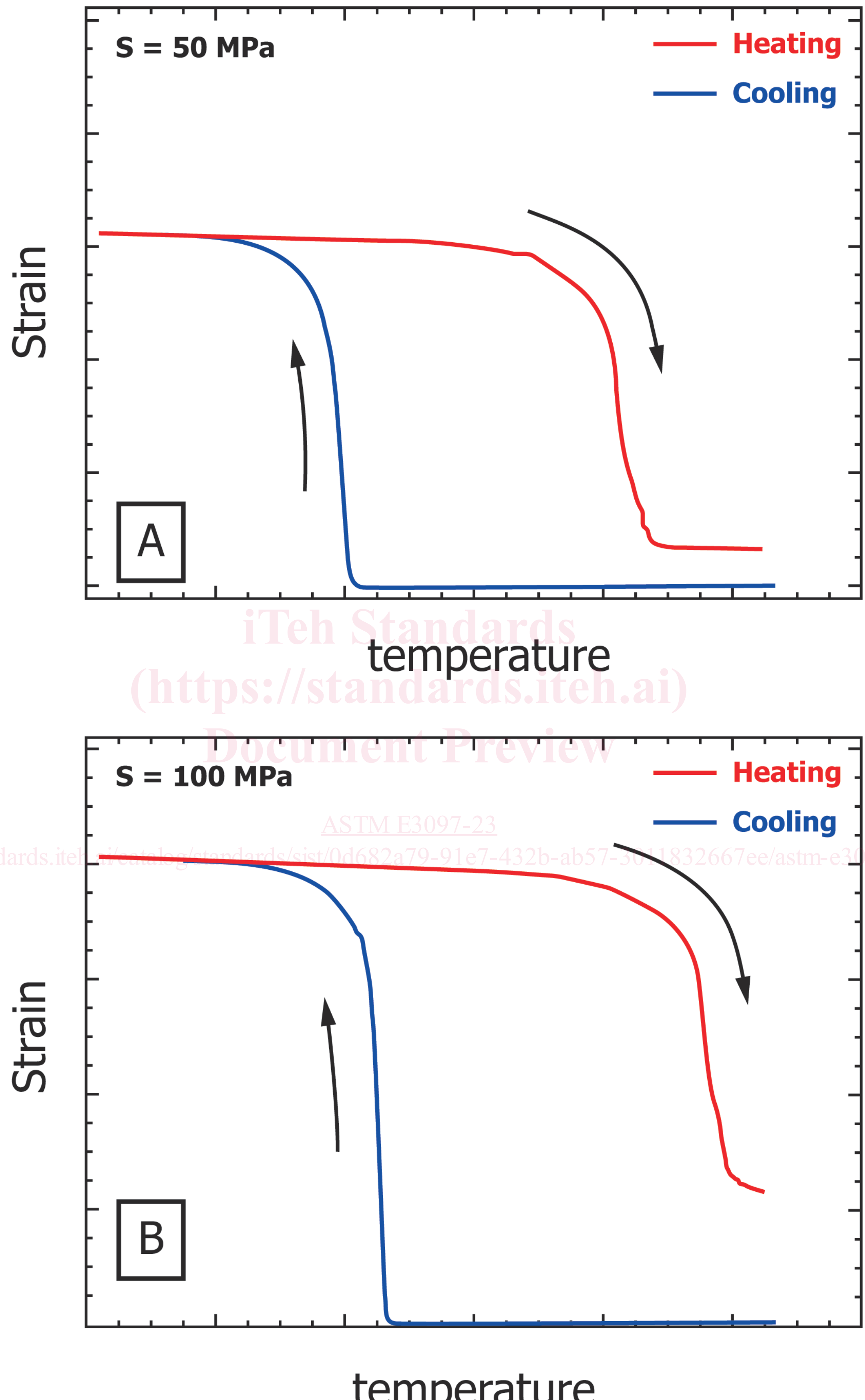

FIG. 2 Effects of Force and Upper Cycle Temperature on test results.
A) UCT sufficient for complete Austenitic transformation. B) UCT not sufficient for complete Austenitic transformation.