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Standard Test Method for Tension Testing of Nickel-Titanium Superelastic Materials[1]

This standard is issued under the fixed designation F2516; 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 the tension testing of superelastic nickel-titanium (nitinol) materials, specifically the methods for determination of upper plateau strength, lower plateau strength, residual elongation, tensile strength, and elongation.

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, 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]

E6 Terminology Relating to Methods of Mechanical Testing
E8/E8M Test Methods for Tension Testing of Metallic Materials
E83 Practice for Verification and Classification of Extensometer Systems
E111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1876 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio by Impulse Excitation of Vibration
E3098 Test Method for Mechanical Uniaxial Pre-strain and Thermal Free Recovery 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/F2082M Test Method for Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery

3. Terminology

3.1 The definitions of terms relating to tension testing appearing in Terminology E6 and the terms relating to nickel-titanium shape

[1] This test method is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.15 on Material Test Methods.

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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.

memory alloys appearing in Terminology F2005 shall be considered as applying to the terms used in this test method. Engineering stress and strain are assumed unless otherwise noted. Additional terms being defined are as follows (see Fig. 1):

3.2 *Definitions:*

3.2.1 *alignment stress, n*—stress (not to exceed 7 MPa) applied to the specimen after it is installed in the grips to ensure that the specimen is straight and aligned to the grips.

3.2.2 *elongation at fracture* (~~El_F~~ El_{fr}), *n*—elongation measured just prior to the sudden decrease in force associated with fracture. See Fig. 1 and X1.2. **E6**

3.2.2.1 *Discussion*—
Elongation at fracture results may be very sensitive to test variables such as test speed, specimen geometry, heat dissipation, surface finish, and alignment. See Test Methods E8/E8M.

3.2.2.2 *Discussion*—
Corrections for ~~non-uniform~~nonuniform strains between the extensometer attachments, including in the necked region, are beyond the scope of this standard. See Test Methods E8/E8M.

3.2.3 *fracture ductility* (~~ε_f~~ ε_{fr}), *n*—true plastic strain at fracture. See X1.2. **E6**

3.2.3.1 *Discussion*—
For prismatic specimens, the fracture ductility is calculated as follows:

~~$$\varepsilon_f = \ln\left(\frac{A_O}{A_f}\right) = \ln\left(\frac{1}{1 - RA\%/100}\right) \quad (1)$$~~

$$\varepsilon_{fr} = \ln\left(\frac{A_O}{A_{fr}}\right) = \ln\left(\frac{1}{1 - RA\%/100}\right) \quad (1)$$

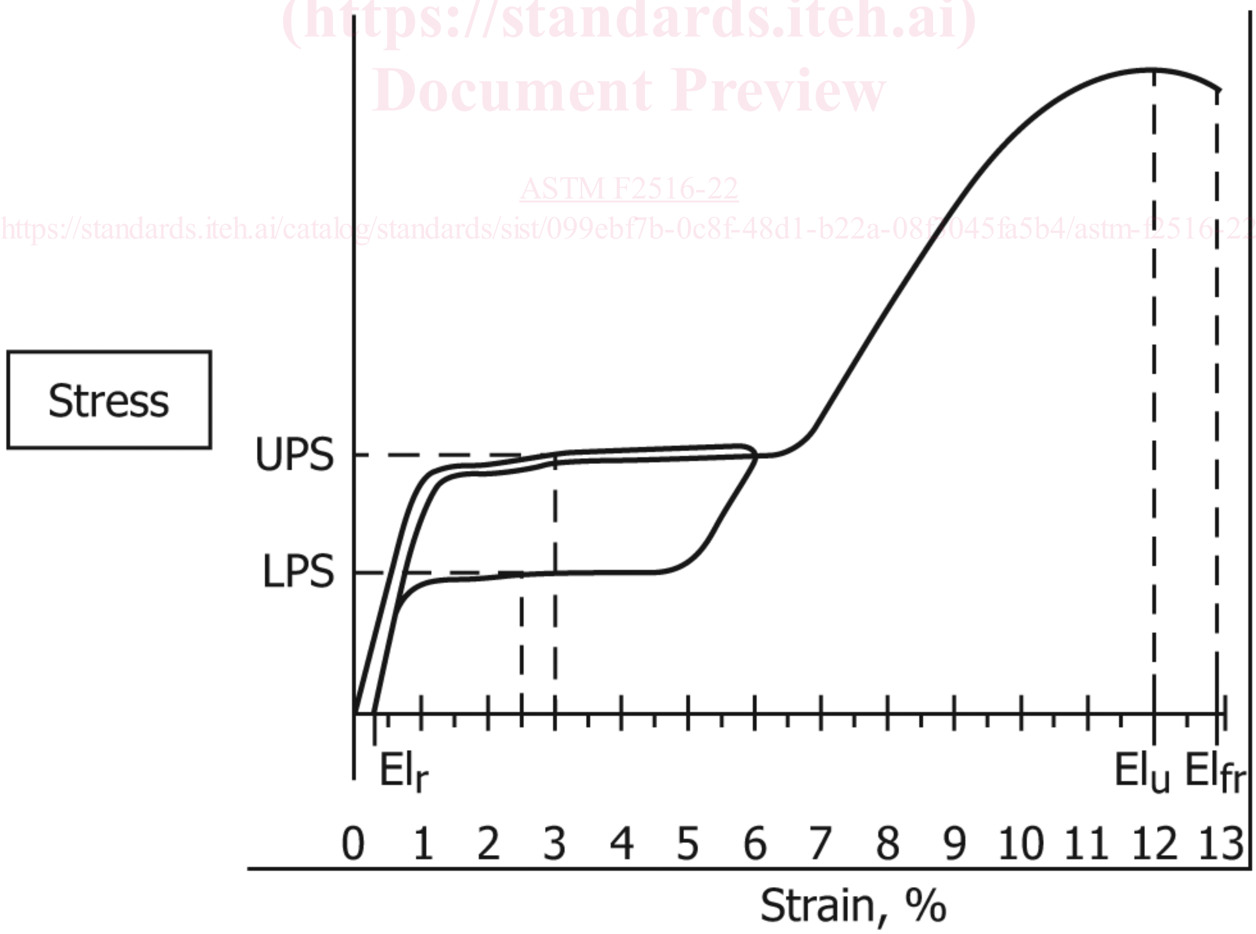

FIG. 1 Terms Illustrated on Typical Stress-Strain Diagram of Superelastic Nitinol

where:

A_O = original cross-sectional area,
A_f = area at fracture of its smallest cross section after testing, and
A_{fr} = area at fracture of its smallest cross section after testing, and
$RA\%$ = reduction of area, %. See Terminology E6.

3.2.4 *lower plateau strength (LPS), n*—stress at 2.5 % strain during unloading of the sample, after loading to 6 % strain. See Fig. 1. **E6**

3.2.5 *reduction of area percent (RA%), n*—percent difference between the original cross-sectional area of a tension test specimen and the area of its smallest cross section after fracture.

3.2.5.1 *Discussion*—
When the specimen necks prior to fracture, reduction in area provides a measure of the material ductility. The reduction of area of a prismatic specimen is calculated using the difference in the original cross-sectional area, A_O, of a specimen and the area at fracture of its smallest cross section, A_{ffr}, after testing as follows:

$$RA\% = 100\% \frac{A_O - A_f}{A_O} \quad (2)$$

$$RA\% = 100\% \frac{A_O - A_{fr}}{A_O} \quad (2)$$

3.2.5.2 *Discussion*—
For measuring a specimen's A_{ffr} with an original circular or rectangular cross sections, see Test Methods E8/E8M, Sectionsubection 7.12.

3.2.6 *residual elongation, El_r, %*—difference between the strain at a set stress at or between the alignment stress and a maximum of 7 MPa during unloading and the strain at that same set stress during the initial loading. See Fig. 1 and X1.4.

3.2.7 *uniform elongation, El_u, %*—elongation determined at the maximum force sustained by the test piece just prior to necking, or fracture, or both. See Fig. 1.

3.2.7.1 *Discussion*—
Uniform elongation is not an accurate measure of ductility. See X1.2.

3.2.8 *upper plateau strength (UPS)*—stress at 3 % strain during loading of the sample. See Fig. 1. **E6**

4. Summary of Test Method

4.1 Using conventional tensile testing apparatus, the material is pulled to 6 % strain, then unloaded to less than 7 MPa, then pulled to failure.

5. Significance and Use

5.1 Tension tests provide information on the strength and the elastic and plastic properties of materials under uniaxial tensile stresses.

5.2 Tension tests, as described in this test method, also provide information on the superelasticity, as defined in Terminology F2005, of the material at the test temperature.

6. Apparatus

6.1 Apparatus is as described in Test Methods E8/E8M.

7. Test Specimen

7.1 Test specimens are as described in Test Methods E8/E8M.

8. Procedure

8.1 Procedure shall be per Test Methods E8/E8M with the following additions:

8.1.1 The material should be tested at a temperature that is a minimum of ~~5°C~~5 °C above the austenitic finish transformation temperature (A_f) in order to prevent testing of a partially transformed material. The temperature of the test should be 22.0 ± ~~2.0°C~~2.0 °C or 37 ± ~~2.0°C,~~2.0 °C, unless otherwise specified. See Terminology F2005 for the definition of A_f. See Test Methods F2004, F2082/F2082M, and E3098 to determine A_f.

8.1.2 The free-running crosshead speed shall be limited per Table 1. See X1.3.

8.1.3 The test shall consist of zeroing the test machine per Test ~~Method~~Methods E8/E8M, gripping the specimen, loading the specimen to the alignment stress, pulling the specimen to 6 % strain, reversing the motion to unload the specimen to the alignment stress, and then pulling the specimen to failure. See X1.4.

8.1.4 For materials with diameter greater than 0.2 mm, strain shall be determined by use of a calibrated extensometer of ~~class~~Class C or better (see Practice E83). For materials with diameter less than or equal to 0.2 mm, strain may be determined by use of an extensometer or by crosshead motion. When using crosshead motion to calculate strain, the length between the grips shall be a minimum of 150 mm. See X1.5.

NOTE 1—Strain should be measured using extensometer versus crosshead displacement, as this will result in a more accurate measurement of strain.

8.1.4.1 When using a clip-on extensometer with small-diameter wire, care shall be taken not to bend or distort the wire when attaching the extensometer.

8.1.5 Upper plateau strength shall be determined as the value of the stress at a strain of 3.0 % during the initial loading of the specimen.

8.1.6 Lower plateau strength shall be determined as the value of the stress at a strain of 2.5 % during the unloading of the specimen.

8.1.7 Residual elongation shall be determined by the difference between the strain at a set stress at or between the alignment stress and a maximum of 7 MPa during unloading and the strain at that same set stress during the initial loading.

NOTE 2—Slippage of the blades of a mechanical contact extensometer can cause variations in the measurement of residual elongation. These errors may be prevented by using O-ring blades or other modifications that increase the contact friction between the mechanical contact extensometer and the test specimen.

NOTE 3—If using a mechanical contact extensometer on a tensile test machine, systematic deviations in the measurement of residual elongation may not become apparent in a test method validation. Comparisons of different extensometer types or test results from different labs using the same extensometer may be required to confirm significant measurement deviations due to extensometer blade slippage during tensile testing of smooth-surfaced NiTi specimens.

NOTE 4—Commercially available mechanical extensometers usually cannot be calibrated to measure displacements below 0.02 mm. Depending on gauge length and acceptance criteria, this may give an unacceptable test precision for residual elongation.

TABLE 1 Crosshead Speed Limits

d, diameter or thickness (mm)[A]	Maximum crosshead speed in mm/min per mm of initial length of reduced section (or initial distance between grips for specimens not having reduced sections)	
	First Cycle (load to 6 % strain and unload)	Second Cycle (load to failure)
$d \leq 0.2$	0.08	0.8
$0.2 < d \leq 0.5$	0.04	0.4
$0.5 < d \leq 2.5$	0.02	0.2
$d > 2.5$	0.01	0.1

[A] For tubing, use *d* that gives an equivalent surface area to diameter ratio; for round tubing, *d* = (outer diameter) – (inner diameter).

8.1.8 The uniform elongation shall be determined by elongation when the maximum force is reached just prior to necking or fracture, or both.

8.1.9 The elongation at fracture shall be determined by elongation measured just prior to the sudden decrease in force associated with fracture.

9. Report

9.1 The report shall include the following information, unless otherwise specified:

9.1.1 Material and sample identification,

9.1.2 Specimen type,

9.1.3 Upper plateau strength,

9.1.4 Lower plateau strength,

9.1.5 Residual elongation,

9.1.6 Tensile strength,

9.1.7 Uniform elongation, if required,

9.1.8 Elongation at fracture,

9.1.9 Test temperature,

9.1.10 Strain determination method (extensometer or crosshead),

9.1.11 Crosshead speed, and

9.1.12 ~~Gage~~Gauge length (length of reduced section or distance between grips for specimens not having reduced sections).

10. Precision and Bias[3]

10.1 An interlaboratory study was conducted in accordance with Practice E691 using three different diameters of superelastic wire. For wire diameters of 0.2 and 0.5 mm, eleven laboratories participated in the study with each laboratory obtaining three results for each diameter. For the 2.5 mm diameter wire, eight laboratories participated in the study with each laboratory obtaining three results. The details are given in ASTM Research Report No. RR:F04-1010.[3]

10.2 The results are summarized in Tables 2-6 for each tensile parameter. The terms *repeatability limit* and *reproducibility limit* are used as specified in Practice E177.

10.3 No measurement of bias is possible with this test method since there is presently no accepted reference material.

TABLE 2 Precision of Upper Plateau Strength (MPa)

Diameter (mm)	Grand Mean	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
0.2	499	13	55	36	154
0.5	492	11	35	30	98
2.5	500	13	25	35	71

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F04-1010. Contact ASTM Customer Service at service@astm.org.

TABLE 3 Precision of Lower Plateau Strength (MPa)

Diameter (mm)	Grand Mean	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
0.2	196	10	35	27	97
0.5	146	9	27	26	75
2.5	138	13	19	36	52

TABLE 4 Precision of Residual Elongation (%)

Diameter (mm)	Grand Mean	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
0.2	0.11	0.09	0.13	0.24	0.36
0.5	0.07	0.03	0.04	0.09	0.10
2.5	0.11	0.05	0.12	0.13	0.33

TABLE 5 Precision of Ultimate Tensile Strength (%)

Diameter (mm)	Grand Mean	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
0.2	1459	45	135	125	377
0.5	1325	23	43	65	120
2.5	1268	15	15	42	41

TABLE 6 Precision of Uniform Elongation (%)

Diameter (mm)	Grand Mean	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
0.2	11.5	0.7	1.2	2.0	3.5
0.5	12.4	0.4	1.0	1.3	2.8
2.5	13.3	0.6	0.7	1.8	1.9

11. Keywords

11.1 lower plateau strength; nickel titanium; nitinol; residual elongation; shape memory; superelasticity; upper plateau strength

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 Measurement of modulus of elasticity requires very precise measurements beyond the scope of this test method. Test Methods E111 and E1876 address determination of modulus of elasticity. For superelastic nitinol, the dynamic method (Test Method E1876) is preferred. Note that the modulus of elasticity exhibits large variation with the martensitic transformation (**1**).[4]

X1.2 Tensile loading of nickel-titanium superelastic materials to fracture will generally encompass elastic strains, phase transformation strains, martensite reorientation strains ~~(twinning)~~(twinning), and plastic strains (**2**). Tensile loading following plastic yielding and the accompanying drop in tangent ~~modulus,~~modulus usually results in an instability that localizes the highest stress into a narrowed neck region and ends in fracture. The ~~Uniform Elongation~~uniform elongation measurement, which is determined at the onset of necking, is not an accurate measure of ductility. However, ~~Uniform Elongation~~uniform elongation can be useful as a process control measure. Elongation at ~~Fracture~~fracture provides an overall elongation value that includes elongation between the extensometer attachment points, including the necking elongation, but is dependent on the ~~gage~~gauge length and underestimates the strain in the necked fracture region. Conventionally, ~~Reduction~~reduction of ~~Area~~area and ~~Fracture Ductility~~fracture ductility are used to estimate the high strains in the necked region. They are interpreted as measures of material ductility, the ability of a material to deform plastically before fracturing (see Terminology E6). In addition, by measuring the longitudinal shape of the neck, an estimate of the true stress at fracture may be calculated (**3**).

[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.

X1.3 During tensile testing of superelastic nitinol material, heat is given off during the austenite-to-martensite transformation. Strain rate is limited to allow the heat to transfer out of the specimen. Otherwise, the increase in specimen temperature will influence the stress-strain response (**4**).

X1.4 Due to experimental problems associated with the establishment of the origin of the stress-strain curve, such as mechanical backlash, initial grip alignment, and specimen curvature, residual elongation may be negative at zero force. In addition, force transducers are typically not calibrated at zero force. For these reasons, the residual elongation is measured while there is a small stress of 7 MPa on the sample.

X1.5 Use of crosshead motion to calculate strain is allowed for small wires due to the possibility of distorting the wire with clip-on type extensometers. In this case, a minimum length between grips is specified to minimize elongation errors due to deflection of the testing equipment. Another alternative is to use a ~~non-contacting~~noncontacting video extensometer.

X2. INTERLABORATORY TEST RESULTS

X2.1 The details of the interlaboratory study are given in ASTM Research Report No. RR:F04-1010. The data used to generate the precision statistics are charted below in Figs. X2.1-X2.5.

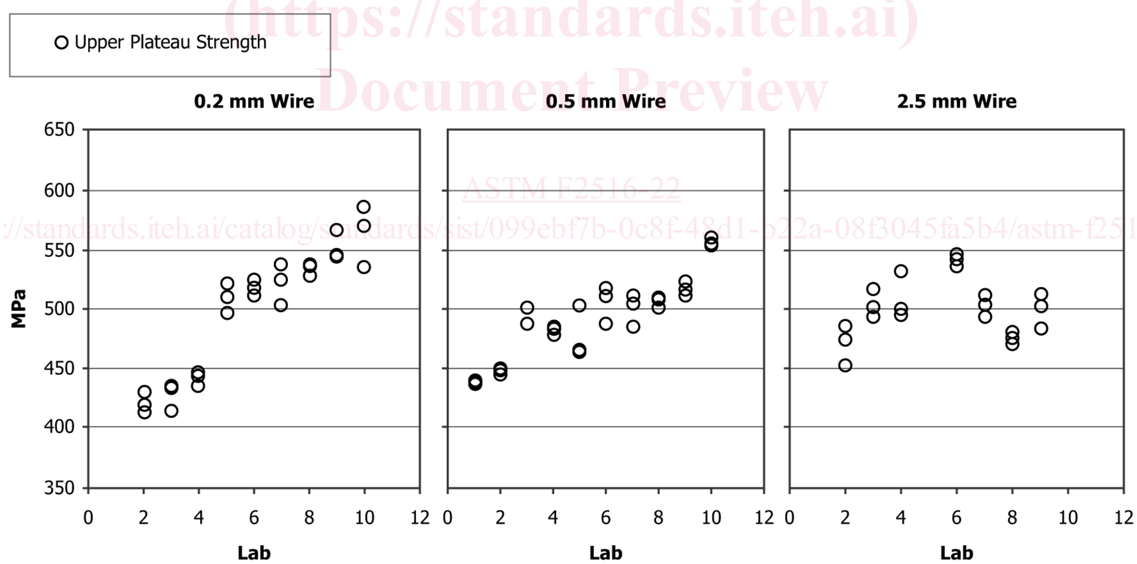

FIG. X2.1 Upper Plateau Strength