Designation: F2516 – 14

# 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 and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

E6 Terminology Relating to Methods of Mechanical Testing

E8 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

F2005 Terminology for Nickel-Titanium Shape Memory Alloys

## 3. Terminology

3.1 The definitions of terms relating to tension testing appearing in Terminology E6 and the terms relating to nickel-titanium shape memory alloys appearing in Terminology F2005 shall be considered as applying to the terms used in this test method. Additional terms being defined are as follows (see Fig. 1):

3.2 *Definitions:*

3.2.1 *lower plateau strength (LPS)*—the stress at 2.5 % strain during unloading of the sample, after loading to 6 % strain.

3.2.2 *residual elongation, $El_r$[%]* —the difference between the strain at a stress of 7.0 MPa during unloading and the strain at a stress of 7.0 MPa during loading.

3.2.3 *uniform elongation, $El_u$[%]*—the elongation determined at the maximum force sustained by the test piece just prior to necking, or fracture, or both.

3.2.4 *upper plateau strength (UPS)*—the stress at 3 % strain during loading of the sample.

## 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 ductility 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.

## 7. Test Specimen

7.1 Test specimens are as described in Test Methods E8.

## 8. Procedure

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

[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.

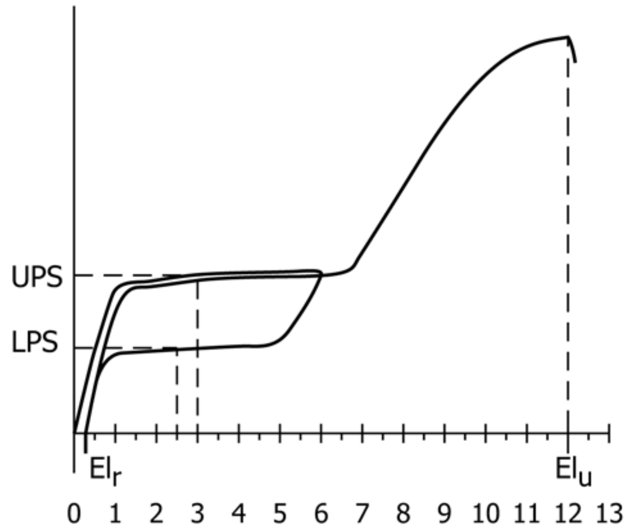


**FIG. 1 Terms Illustrated on Typical Stress-Strain Diagram of Superelastic Nitinol**

8.1.1 Unless otherwise specified, the temperature of the test shall be 22.0 ± 2.0°C. It is recommended that the material be tested at a temperature that is a minimum of 5°C above the austenitic finish transformation temperature ($A_f$) in order to prevent testing of a partially transformed material.

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

8.1.3 The test shall consist of zeroing the force transducer, gripping the specimen, pulling the specimen to 6 % strain, reversing the motion to unload the specimen to less than 7 MPa, and then pulling the specimen to failure.

8.1.4 For materials with diameter greater than 0.2 mm, strain shall be determined by use of a calibrated extensometer of 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 must be 150 mm.

NOTE 1—It is recommended that strain be measured using extensometer versus crosshead displacement as this will result in a more accurate measurement of strain.

**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) |
| $d \le 0.2$ | 0.08 | 0.8 |
| $0.2 < d \le 0.5$ | 0.04 | 0.4 |
| $0.5 < d \le 2.5$ | 0.02 | 0.2 |
| $d > 2.5$ | 0.01 | 0.1 |

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

8.1.4.1 When using a clip-on extensometer with small-diameter wire, care must 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 stress of 7.0 MPa during unloading and the strain at a stress of 7.0 MPa during loading.

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.

## 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,

9.1.8 Test temperature,

9.1.9 Strain determination method (extensometer or crosshead),

9.1.10 Crosshead speed, and

9.1.11 Gage 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 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.

## 11. Keywords

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

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F04-1010.

**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 |

**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 |

# APPENDIXES

**(Nonmandatory Information)**

## X1. RATIONALE

X1.1 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]

X1.2 Measurement of modulus of elasticity requires very precise measurements beyond the scope of this standard. 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.[5]

X1.3 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.

X1.4 Use of crosshead motion to calculate strain is allowed for small wires due to the possibility of distorting the wire with

[4] Shaw, J. A. and Kyriakides, S., "On the Nucleation and Propagation of Phase Transformation Fronts in a NiTi Alloy", *Acta Mater*, Vol 45, No. 2, 1997, pp. 683–700.

[5] Spinner, S. and Rozner, A. G., "Elastic Properties of NiTi as a Function of Temperature", *The Journal of Acoustical Society of America*, Vol. 40, No. 5, 1966, pp. 1009–1015.

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 video extensometer.

## X2. INTERLABORATORY TEST RESULTS

X2.1 The details of the interlaboratory study are given in ASTM Research Report F04–1010. The data used to generate the precision statistics are charted below in Figs. X2.1-X2.5.

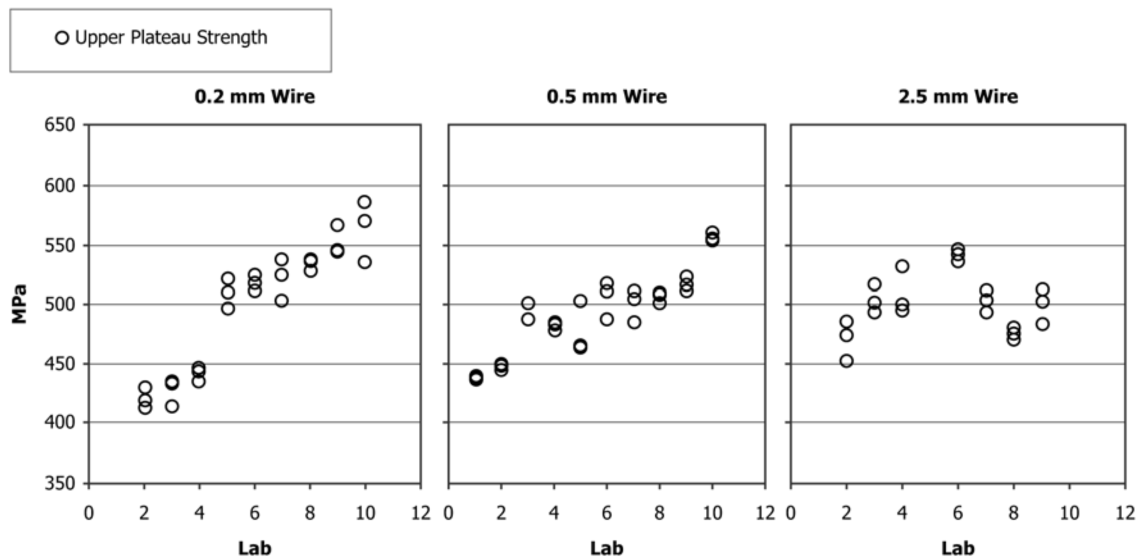


FIG. X2.1 Upper Plateau Strength

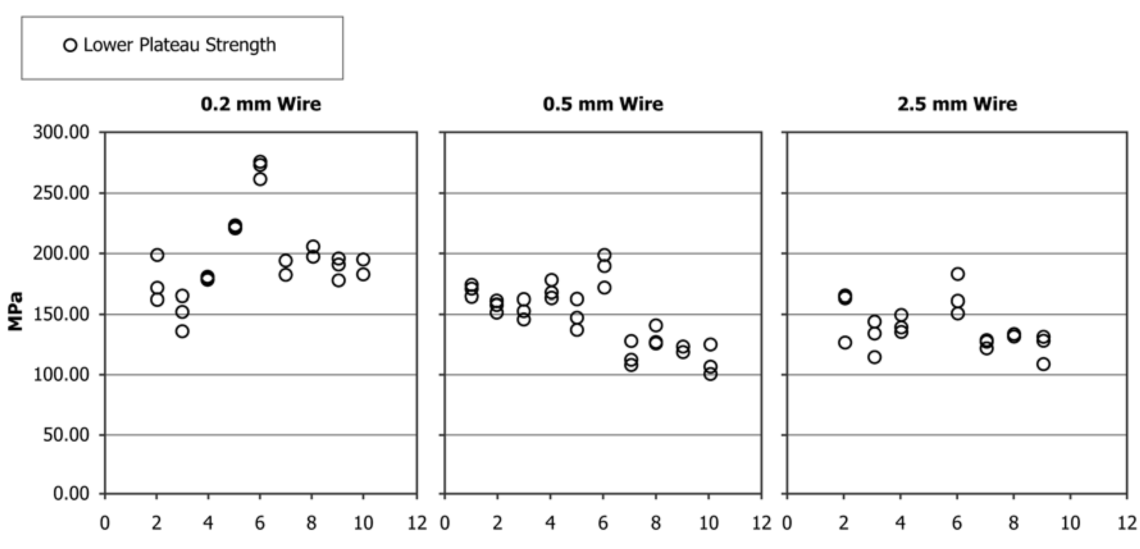


FIG. X2.2 Lower Plateau Strength

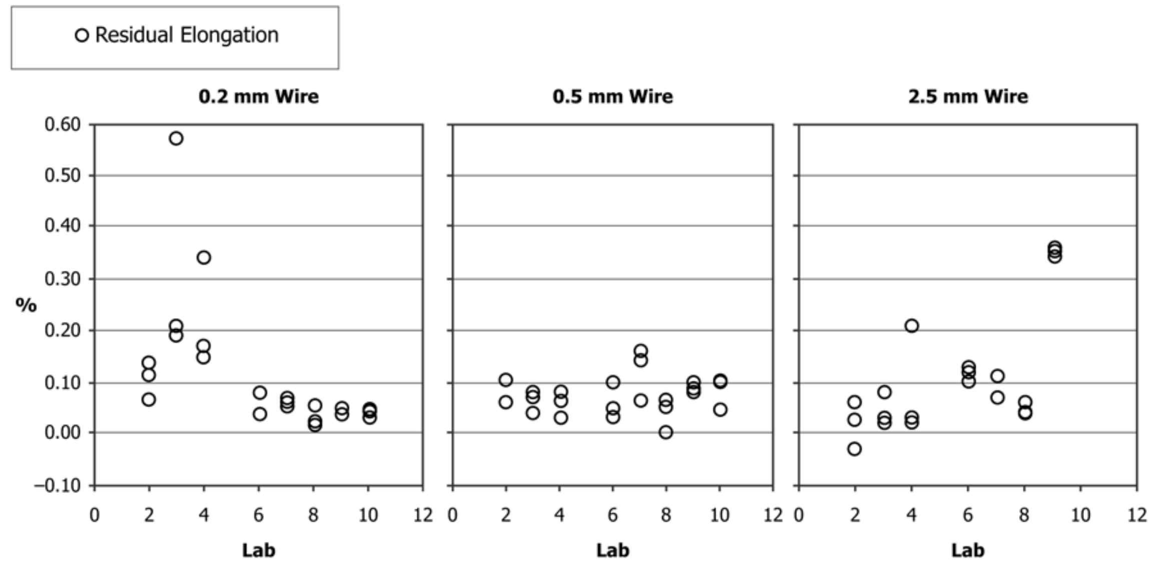


FIG. X2.3 Residual Elongation

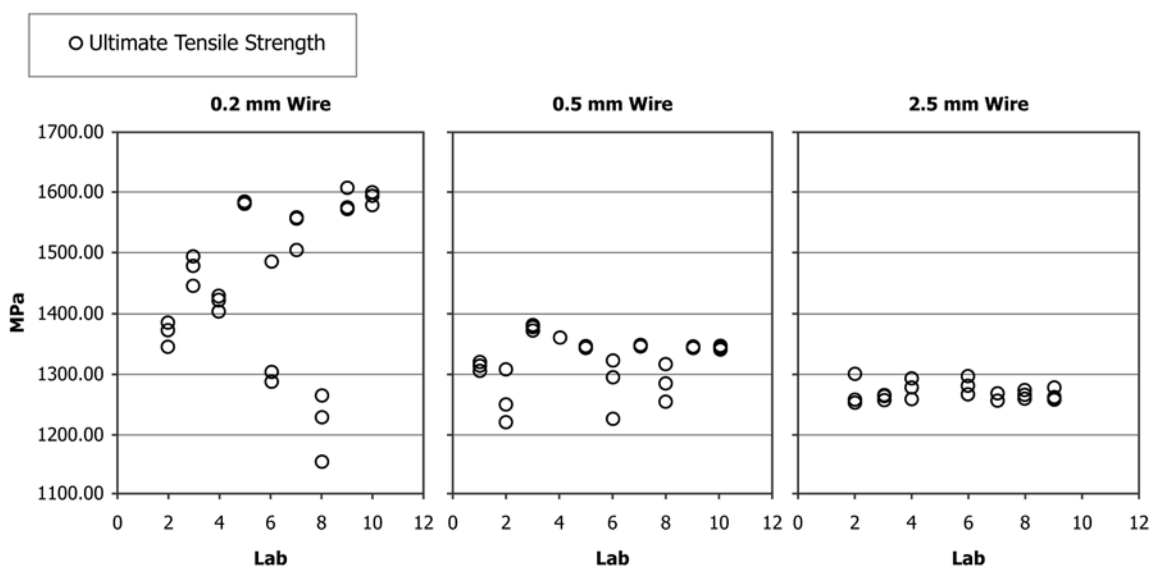


FIG. X2.4 Ultimate Tensile Strength

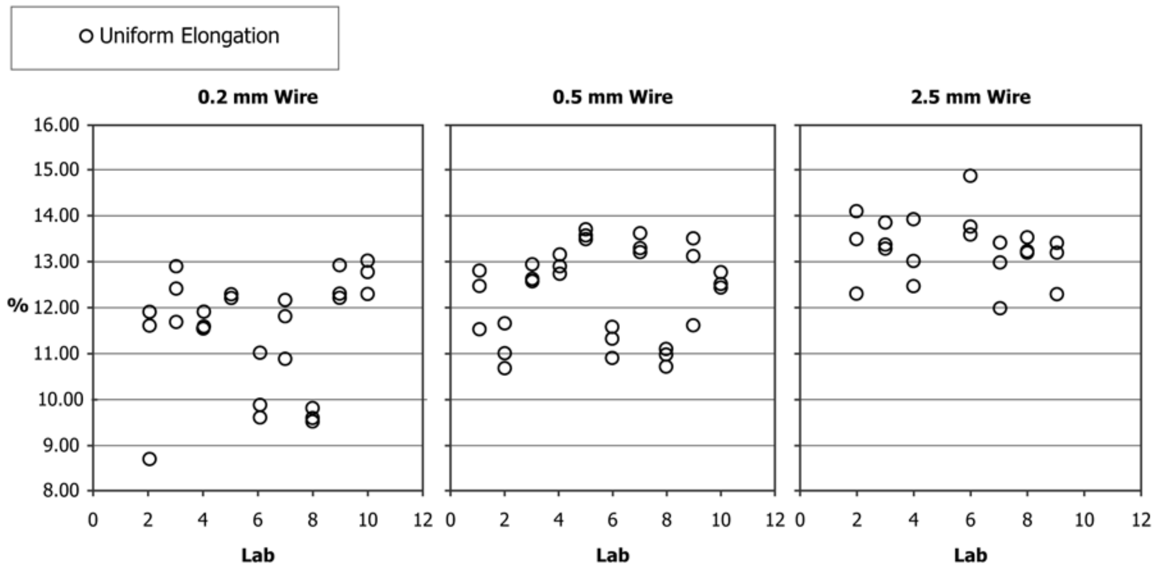


FIG. X2.5 Uniform Elongation