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Standard Test Method for Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery[1]

This standard is issued under the fixed designation F2082/F2082M; 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 a procedure for quantitatively determining the martensite-to-austenite ~~transformation temperatures of either fully annealed or heat-treated nickel-titanium alloys by~~ or the martensite to R-phase transformation temperature of annealed, aged, shape-set, or tempered nickel-titanium alloy specimens by deforming the specimen in bending and measuring the deformation recovered during heating through the thermal ~~transformation.~~ transformation (BFR method). See 3.1.1.

NOTE 1—For aged, shape-set, or tempered specimens the transformation may be from martensite to austenite or from martensite to R-phase. See Reference (**1**)[2] for details.

1.2 The test specimen may be wire, tube, or strip or a specimen extracted from a semifinished or finished component.

1.2.1 For specimens not in the form of a wire, tube, or strip that are extracted from semifinished or finished components, a wire, tube, or strip shaped test specimen shall be made from the component such that the deformation mode in the test specimen is pure bending.

1.2.2 Other specimen geometries or displacements resulting in a more complex strain state, such as bending with torsion or buckling, are beyond the scope of this standard.

1.3 Ruggedness tests have demonstrated that sample A_f must be limited to obtain good test results. See 5.6 for details. Ruggedness tests have demonstrated that deformation strain, deformation temperature, and equilibration time at the deformation temperature must be controlled to obtain good test results. See 9.1, 9.2, and 9.4 for details.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in ~~non-conformance~~nonconformance with this standard.

1.5 *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.*

1.6 *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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E220 Test Method for Calibration of Thermocouples By Comparison Techniques
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
F2004 Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis
F2005 Terminology for Nickel-Titanium Shape Memory Alloys

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Definitions*—Specific technical terms used in this test method are found in Terminology F2005.

3.1.2 *free ~~recovery—~~recovery, n*—unconstrained motion of a shape memory alloy specimen upon heating and transformation to austenite or R-phase after deformation ~~in a lower temperature phase.~~at a temperature below the temperature for the start of the formation of martensite, M_s.

3.1.3 $A_{f\text{-}95}$—austenite finish temperature of a finished wire, tube, or component measured by bend and free recovery using the 95 percent recoverable deformation methodology.

3.1.4 $A_{f\text{-}tan}$—austenite finish temperature of a finished wire, tube, or component measured by bend and free recovery using the tangent methodology.

3.2 *Abbreviations:*

3.2.1 *LVDT*—linear variable differential transducer.

3.2.2 *RVDT*—rotary variable differential transducer.

4. Summary of Test Method

4.1 This test method involves cooling a test specimen to ~~its nominally fully martensitic phase,~~ a temperature below the start temperature for the reversion to austenite, A_s, or to below the start temperature for the reversion to R-phase, R'_s, if there is an intermediate R-phase, deforming the specimen, and heating the specimen to its fully austenitic phase. During heating, the motion of the specimen is measured and plotted versus the specimen temperature. For a two-stage transformation, the ~~R^1~~R'_s,~~R^1~~R'_f,A_s, and A_f, as defined in Terminology F2005, are determined using the tangent methodology. For a single-stage transformation, the A_s and A_f are determined using the tangent methodology. Alternatively, for either single or two-stage transformation material, the A_f may be measured using the 95 percent recoverable deformation methodology.

5. Significance and Use

5.1 This test method provides a rapid, economical method for determination of transformation temperatures.

5.2 Measurement of the specimen 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 uses a wire, tube, ~~or strip samples; thus, it is able to provide~~ strip specimen, or a wire, tube, or strip specimen extracted from a component; thus, it provides an assessment of ~~the~~ a nickel titanium product in its semifinished or finished form.

5.4 This test method may be used on annealed samples to determine the transformation temperatures and ~~assure~~ensure the alloy formulation, since chemical analysis is not precise enough to ~~determine~~ adequately determine the nickel-to-titanium ratio of shape memory alloys.

[3] 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.

5.5 ~~Transformation temperatures derived from this test method may differ from those derived from other methods as a result of~~ In general, the transformation temperatures measured by this method will not be the same as those measured by the DSC method defined in Test Method F2004~~the effects of strain and load on the transformation temperature.~~. Therefore, the results of DSC and BFR cannot be compared directly.

5.5.1 The BFR method measures the transformation temperatures by tracking shape recovery of stress-induced martensite deformed below the R'_s temperature or the A_s temperature. In contrast, the DSC method measures the start, peak, and finish temperatures of the thermal transformation of martensite to R-phase or to austenite. See Refs (**1-4**).

5.6 The test method is applicable to shape memory alloys with A_f temperatures in the range of approximately –25 to ~~± 90°C.~~90 °C.

6. Apparatus

6.1 *LVDT,* with range greater than half the mandrel diameter (see 9.2), with power supply, mounted in an appropriate fixture with counterbalanced probe (see Fig. 1); or RVDT with range greater than 45°, with power supply, mounted in an appropriate fixture (see Fig. 2); or a vision system; or equivalent means of measuring sample displacement.

6.2 *Thermocouple and Indicator,* with resolution of ~~0.1°C (0.2°F)~~0.1 °C [0.2 °F] or better.

6.3 *XY Chart Recorder,* or equivalent manual or automated data acquisition system.

6.4 *Hot Plate and Stirrer.*

6.5 *Bath of Heat Transfer Fluid,* for example, denatured alcohol, ethylene glycol, ~~water, and so forth.~~or water, or a fluid agreed upon between the customer and supplier.

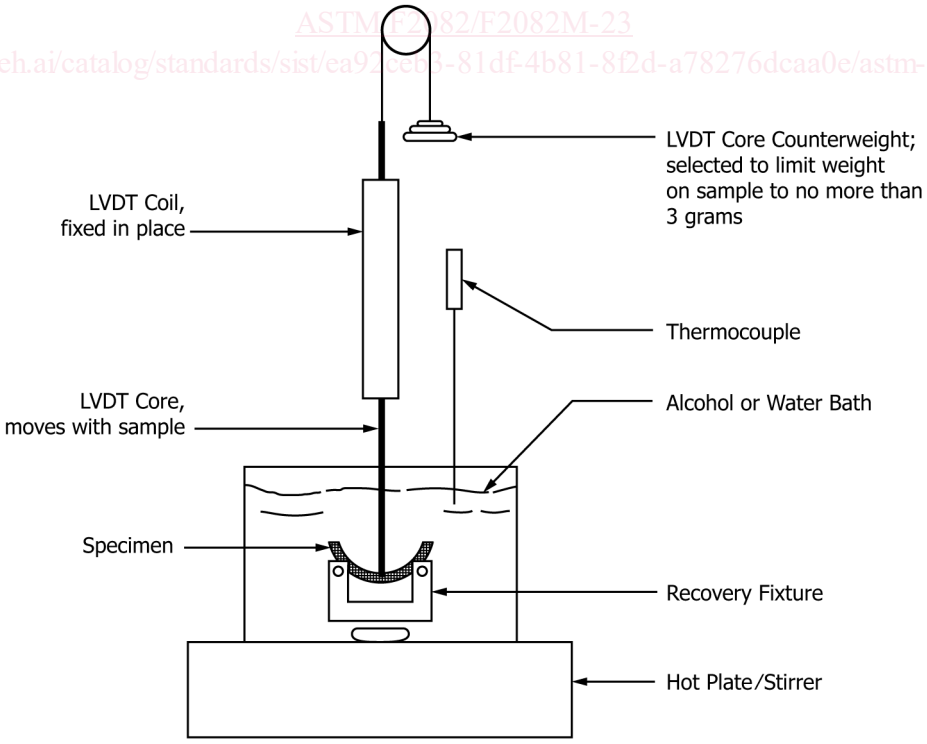

FIG. 1 Schematic Showing Side View of Test Apparatus Using a Vertically Mounted and Counterbalanced LVDT (LVDT Power Supply, Thermocouple Indicator, and Data Acquisition System are not shown)

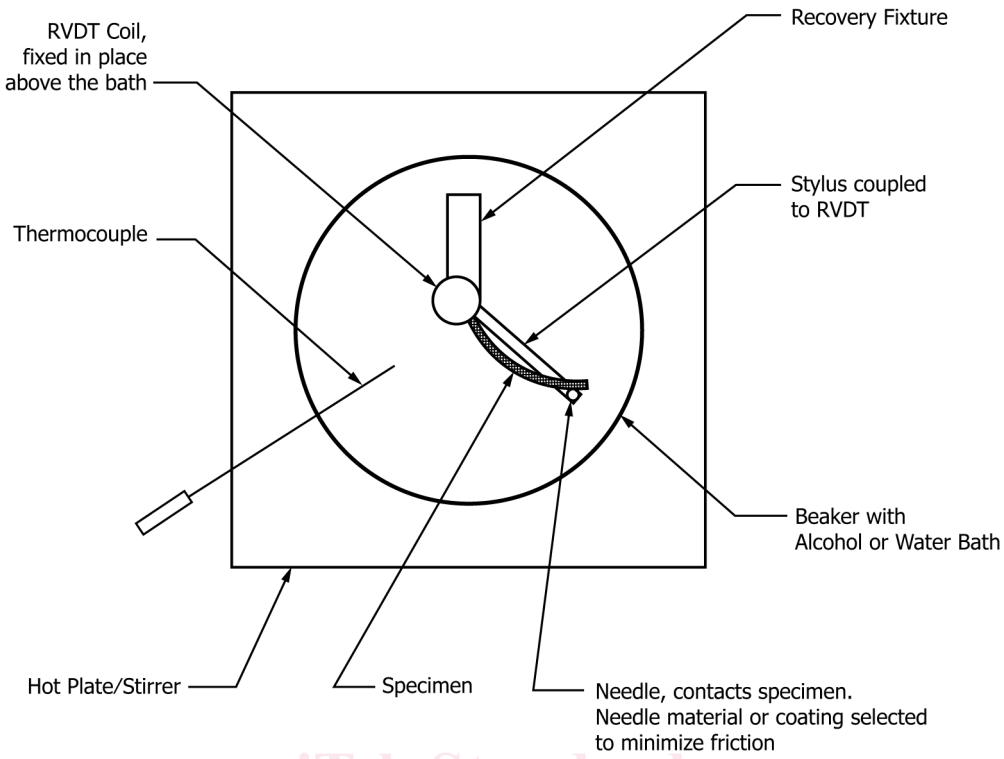

FIG. 2 Schematic Showing Top View of Test Apparatus ~~using~~Using an RVDT
(RVDT Power Supply, Thermocouple Indicator, and Data Acquisition System are not shown)

6.6 *Mandrel,* for deforming the sample in the martensitic state.

6.7 *Fixture,* for holding the sample during recovery.

6.8 *Liquid Nitrogen,* or dry ice.

7. Sampling

7.1 ~~Test specimen can~~The test specimen shall be a wire, tube, or ~~strip with diameter or thickness in the range of 0.3 to 3.0 mm (0.012 to 0.12 in.). For test systems that do not contact the specimen (for example, vision system), the~~ strip, or a wire, tube, or strip specimen extracted from a component with the specimen diameter or thickness ~~may be less than 0.3 mm.~~in the range of 0.3 to 3.0 mm [0.012 to 0.12 in.].

7.1.1 For test systems that do not contact the specimen (for example, vision system), the diameter or thickness of the specimen may be less than 0.3 mm.

7.2 Specimens ~~may~~shall be tested in the ~~semifinished (heat-treated) or annealed condition. Anneal is~~ annealed, aged, shape-set, or tempered condition as defined in Terminology F2005~~.~~ and required by the product specification.

8. Calibration

8.1 The thermocouple and indicator shall be kept in a calibrated condition, traceable to the National Institute for Standards and Technology or appropriate National Metrology Institute that successfully participates in relevant international interlaboratory comparisons.

8.2 The thermocouple shall be calibrated using Test Method E220.

9. Procedure

9.1 For alloys that are ~~superelastic~~austenitic at room temperature, cool a bath of appropriate heat transfer fluid to ~~–55°C (–67°F)~~ –55 °C [–67 °F] or lower using liquid nitrogen, dry ice, or other suitable method. For alloys that are martensitic or R-phase at room temperature, cool the bath to ~~10°C (50°F)~~10 °C [50 °F] or lower.

9.2 Select a mandrel according to the sample diameter or thickness to give an outer fiber strain of 2 to 2.5 %. For these strains, mandrel diameter shall be between 39 and 49 times specimen diameter or thickness.

NOTE 2—Outer fiber strain, e%, is calculated as follows: e% (decimal equivalent) = r / (r + R), where r = radius or half thickness of the test specimen and R = radius of the mandrel. See Ref (**5**).

9.3 Cut a test specimen long enough to wrap 90 to 180° around the mandrel.

9.4 Place the recovery fixture and the mandrel, along with the test specimen, in the bath and wait a minimum of 3 min for the ~~fixtures~~ fixture, mandrel, and specimen to equilibrate to the bath temperature.

9.5 Deform the specimen in the bath by wrapping it 90 to 180° around the mandrel.

9.6 Place the specimen on a fixture (recovery fixture) that holds the sample so as not to interfere with the free recovery of the specimen on heating.

9.7 Remove the mandrel from the bath. Alternatively, the mandrel can be attached to the recovery fixture and left in the bath. In this case, the thermal mass of the mandrel and fixture shall be such that the temperature of the fixture and the bath is uniform throughout the test.

9.8 Set the apparatus to measure the motion of the sample.

9.8.1 For an LVDT, lower the LVDT core onto the specimen as shown in Fig. 3. The weight of the LVDT core shall be counterbalanced such that the weight on the specimen is no more than 3 g.

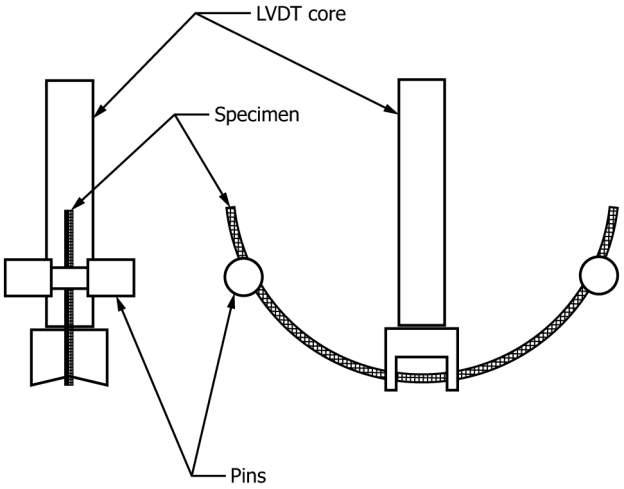

FIG. 3 Placement of LVDT Core on Deformed Specimen, ~~which~~Which is resting on Recovery Fixture Pins

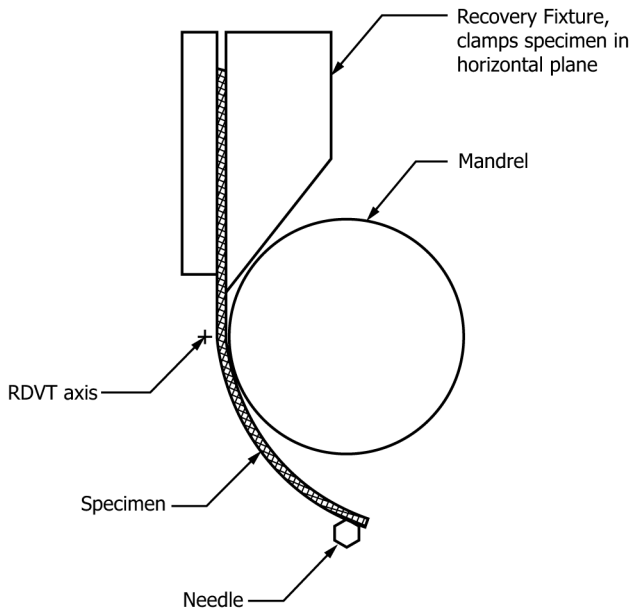

FIG. 4 Placement of Needle on Deformed Specimen, ~~which~~Which is Clamped to the Recovery Fixture. Top View Shown with Stylus and RVDT Removed. Note ~~that~~That the Recommended RVDT Axis Location is Offset from the Mandrel by the Radius of the Needle

9.8.2 For an RVDT, make sure that the needle is in contact with the test specimen (Fig. 4). To minimize friction effects, the needle shall be encased in a polytetrafluoroethylene (PTFE) sheath, or the needle shall be constructed from or coated with PTFE or material with equivalent friction.

9.9 Place the thermocouple in the bath as close to the specimen as is practical.

9.10 Set the *XY* chart or data acquisition system to record the temperature on the either *X* or *Y* axis and sample motion on the other axis.

9.11 Stir and heat the bath on the hot plate to a temperature above the A_f (measured according to method to be used in Section 10). Limit the heating rate to no more than ~~4°C/min~~4 °C/min during the recovery.

9.12 Stop the test once the temperature is at least ~~10°C~~10 °C above the A_f (measured according to method to be used in Section 10), as determined by noting that the sample is straight and the displacement-versus-temperature curve has flattened. Turn off the hot plate and stop recording.

10. Determination of Transformation Temperature

10.1 Determine the transformation temperatures (~~R^1~~R'_s, ~~R^1~~R'_f, A_s, or A_f) using the tangent method or determine A_f using the 95 percent recoverable deformation methodology.

10.2 To determine A_s and $A_{f\text{-}tan}$, refer to ~~Fig. 5~~Figs. 5 and 6 ~~and Fig. 6~~. The transformation may occur in one or two stages. For a one-stage transformation, the middle tangent line should be drawn tangent to the steepest portion of the curve (see Fig. 5). In the case of a two-stage transformation, one line should be drawn tangent to the steepest slope observed in the first stage of the transformation, and a second line should be drawn tangent to the steepest slope in the second stage of the transformation (see Fig. 6).

10.3 To determine the $A_{f\text{-}95}$ transformation temperature, refer to Fig. 7. $A_{f\text{-}95}$ is determined when the deformation is 95 % recovered. Although the test may be started at lower temperatures than –55 °C, the deformation of the sample at –55 °C shall be considered to be its fully deformed condition ~~(i.e.,~~ (that is, 0 % recovery). A 100 % recovery of deformation shall be considered to occur ~~10°C~~10 °C beyond the temperature that the displacement-versus-temperature curve attains a constant slope.