Designation: F 2082 – 01

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 F 2082; 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 determining the martensite-to-austenite transformation temperatures of nickel titanium alloys by measuring the deformation recovered during the thermal transformation.

1.2 The values in SI units are to be regarded as the standard. The values given in inch-pound units are provided for information only.

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:*

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques[2]

F 2005 Terminology for Nickel-Titanium Shape Memory Alloys[3]

3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are found in Terminology F 2005.

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, 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'_s, R'_f, A_s, and A_f, as defined in Terminology F 2005, are determined. For a single-stage transformation, the A_s and A_f are determined.

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 wire, tube, or strip samples; thus, it is able to provide an assessment of the product in its semifinished form.

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

5.5 Transformation temperatures derived from this test method may differ from those derived from other methods as a result of effects of strain and load on the transformation temperature.

5.6 The test method is applicable to shape memory alloys with A_f temperatures in the range of approximately –25 to +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).

6.2 *Thermocouple and Indicator*, with resolution of 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 *Denatured Alcohol Bath*, or water bath.

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

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

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[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] *Annual Book of ASTM Standards*, Vol 14.03.

[3] *Annual Book of ASTM Standards*, Vol 13.01.

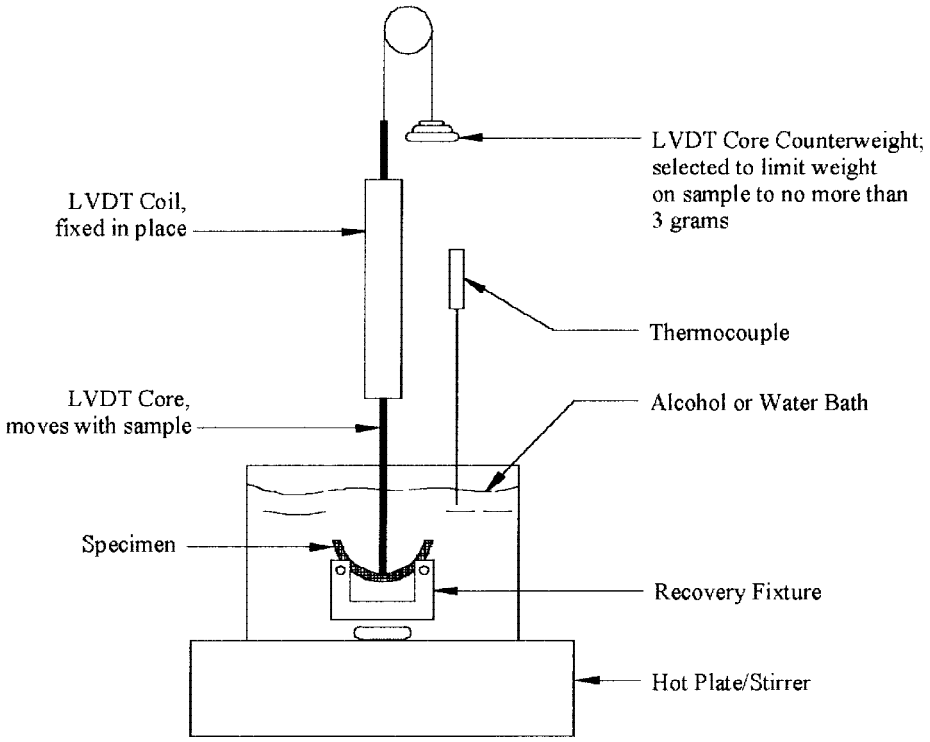

FIG. 1 Schematic Showing Side View of Test Apparatus Using a Vertically Mounted and Counterbalanced LVDT (LVDT Power Supply, Thermocouple Indicator, and Data Acquisition System Are Not Shown)

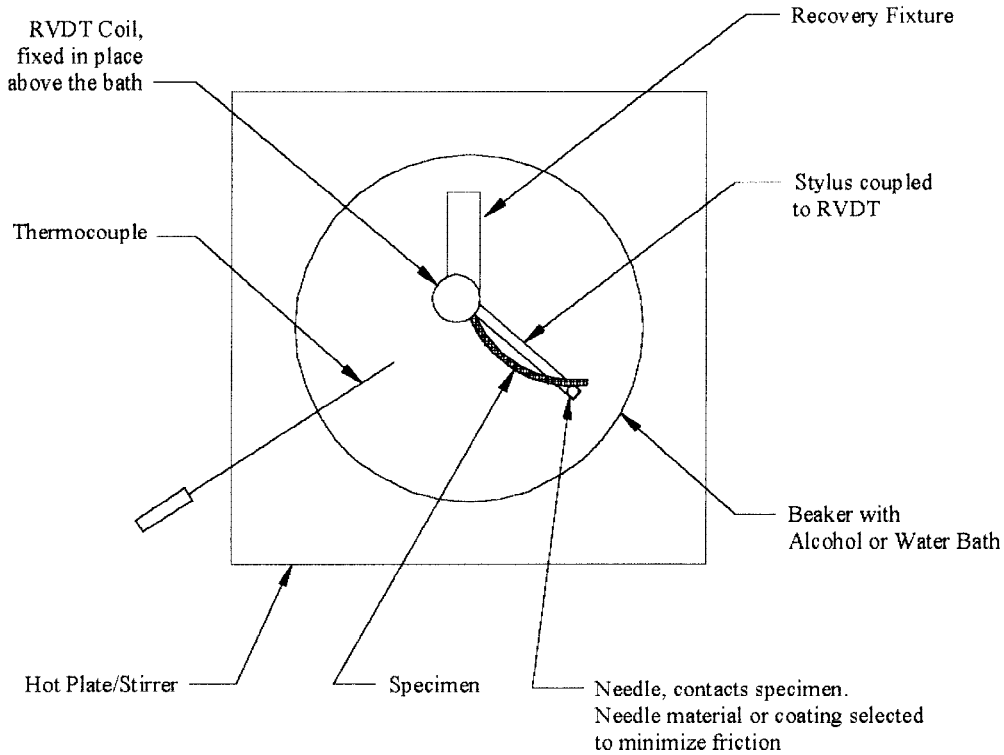

FIG. 2 Schematic Showing Top View of Test Apparatus Using an RVDT (RVDT Power Supply, Thermocouple Indicator, and Data Acquisition System Are Not Shown)

6.8 *Probe or Needle,* for contacting and moving with the sample during recovery.

6.9 *Liquid Nitrogen,* or dry ice.

7. Sampling

7.1 Test specimen can 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.).

7.2 Specimens may be tested in the semifinished (heat-treated) or annealed condition. Anneal is defined in Terminology F 2005.

8. Calibration

8.1 The thermocouple and indicator shall be kept in a calibrated condition, traceable to the National Institute for Standards and Technology.

8.2 The thermocouple shall be calibrated using Test Method E 220.

9. LVDT Procedure

9.1 For alloys that are superelastic at room temperature, cool the alcohol bath to –40°C (–40°F) or lower using liquid nitrogen or dry ice. For alloys that are martensitic at room temperature, cool the water bath to 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 4 %. For these strains, mandrel diameter shall be between 24 and 49 times specimen diameter or thickness.

9.3 Cut a test specimen long enough to wrap 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 2 min for the fixtures to equilibrate to the bath temperature.

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

9.6 Remove the mandrel from the bath.

9.7 Place the deformed specimen on the recovery fixture. For best results, the recovery fixture should support the deformed specimen on two pins spaced at a distance equal to 80 to 95 % of the diameter of the mandrel.

9.8 Set the device to measure the motion of the sample. Lower the LVDT core onto the specimen as shown in Fig. 3. The weight of the LVDT core must be counterbalanced such that the weight on the specimen is no more than 3 g.

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 *X* axis and sample motion on the *Y* axis.

9.11 Stir and heat the bath on the hot plate to a temperature above the A_f. Limit the heating rate to no more than 4°C/min during the recovery.

9.12 Stop the test once the temperature is at least 10°C above the A_f, 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. RVDT Procedure

10.1 For alloys that are superelastic at room temperature, cool the alcohol bath to –40°C (–40°F) or lower using liquid nitrogen or dry ice. For alloys that are martensitic at room temperature, cool the water bath to 10°C (50°F) or lower.

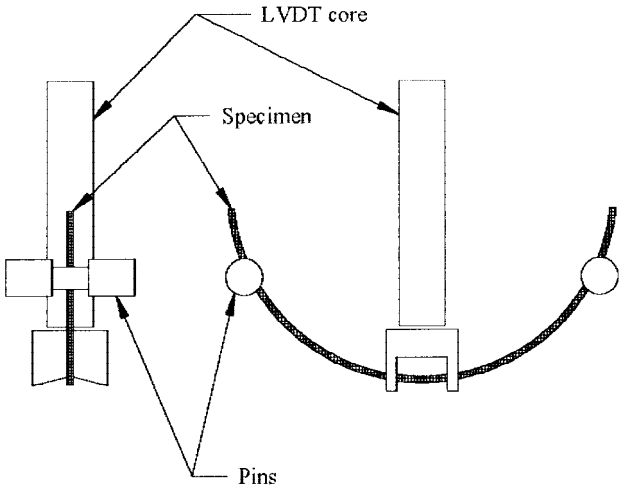

FIG. 3 Placement of LVDT Core on Deformed Specimen, Which Is Resting on Recovery Fixture Pins

10.2 Select a mandrel according to the sample diameter or thickness to give a strain of 2 to 4 %. For these strains, mandrel diameter shall be between 24 and 49 times specimen diameter or thickness.

10.3 Cut a test specimen long enough to wrap 90° around the mandrel, plus a tail for clamping.

10.4 Place the recovery fixture and the mandrel, along with the test specimen, in the bath and wait a minimum of 2 min for the fixtures to equilibrate to the bath temperature.

10.5 Clamp the specimen in the recovery fixture. The fixture shall hold the sample in the horizontal plane.

10.6 Deform the specimen in the bath by wrapping it 90° around the mandrel.

10.7 Remove the mandrel from the bath. Alternately, for the RVDT method, 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 must be such that the temperature of the fixture and the bath is uniform throughout the test.

10.8 Set the device to measure the motion of the sample. Make sure the needle is in contact with the test specimen (Fig. 4). To minimize friction effects, the needle shall be encased in a PTFE sheath, or the needle shall be constructed from or coated with an equivalent material.

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

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

10.11 Stir and heat the bath on the hot plate to a temperature above the A_f. Limit the heating rate to no more than 4°C/min during the recovery.

10.12 Stop the test once the temperature is at least 10°C above the A_f, 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.

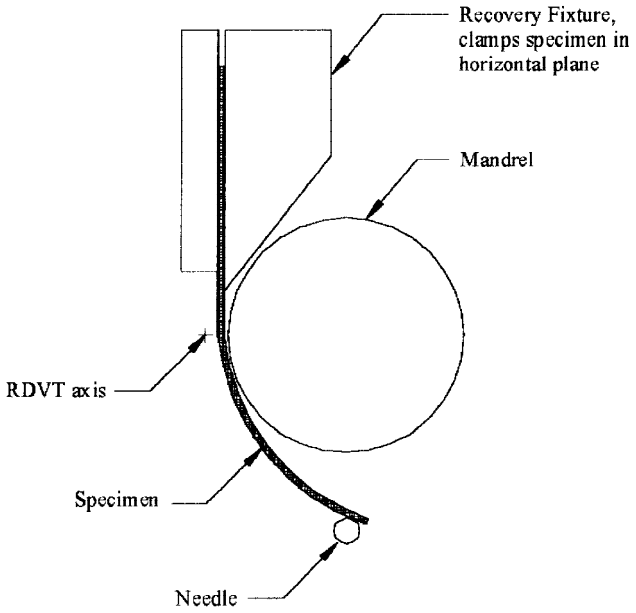

FIG. 4 Placement of Needle on Deformed Specimen, Which Is Clamped to the Recovery Fixture. Top View Shown with Stylus and RVDT Removed. Note the Recommended RVDT Axis Location Is Offset from the Mandrel by the Radius of the Needle

11. Determination of Transformation Temperature

11.1 Determine the transformation temperatures according to Fig. 5 or 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).

12. Report

12.1 The report shall include the following information:

12.1.1 Complete identification of the material tested including specification, lot number, and heat treatment.

12.1.2 Results of the transformation measurements, reported to the nearest 1°C.

13. Precision and Bias

13.1 The precision and bias of this test method is to be established.

14. Keywords

14.1 free recovery; nickel titanium; nitinol; shape memory; transformation temperature

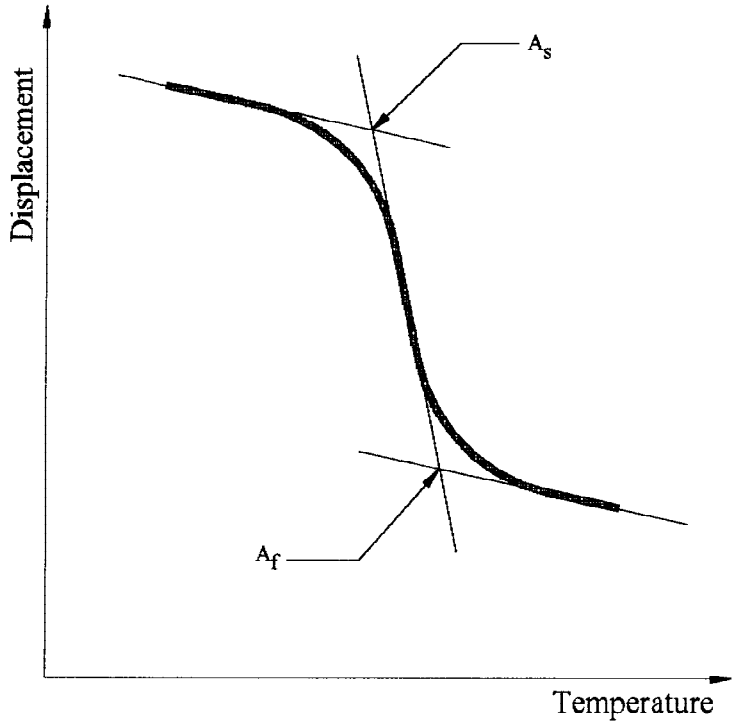

FIG. 5 One-Stage Transformation—Tangent Lines and Transformation Temperatures

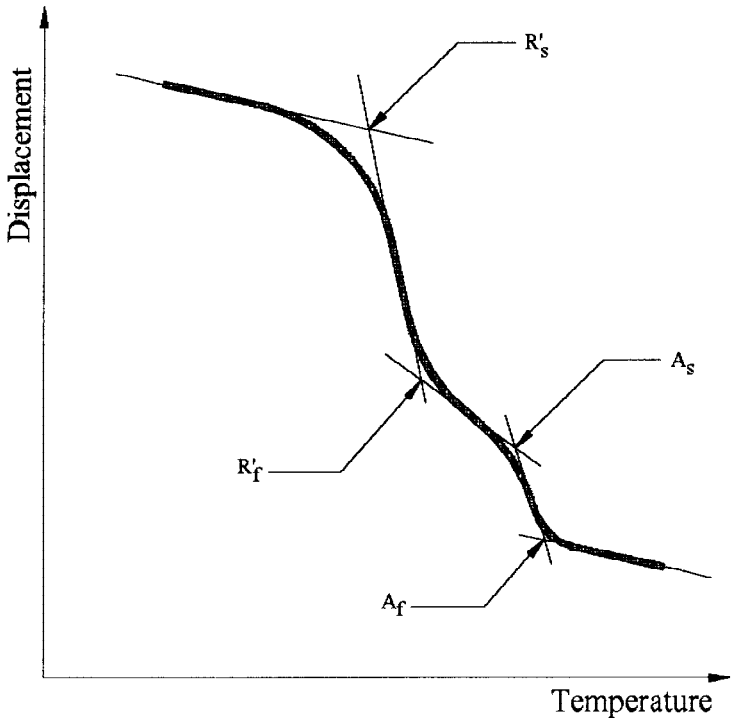

FIG. 6 Two-Stage Transformation—Tangent Lines and Transformation Temperatures

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 Transformation temperature is used to characterize nickel-titanium alloys in the form of raw material, semifinished material, and finished product. In the case of raw material, transformation testing is necessary because chemical analysis is not precise enough to predict the desired shape memory and superelastic properties.

X1.2 This test method provides a rapid, economic means of determining the martensitic-to-austenitic transformation temperatures by recording the motion of the samples as it exhibits the shape memory effect. In the case of finished products, this test method often is used to determine the shape memory behavior of the product in its final application.

X1.3 Transformation temperatures measured by this test method will differ from those measured by thermal analysis or other techniques as a result of the effects of strain and load.

X1.4 A strain level of 2 to 4 % is selected to minimize the effect of strain on the transformation temperatures.

X1.5 The heating rate is limited to minimize the thermal gradients in the bath. The heating rate may be controlled manually or through the use of a temperature controller.

X1.6 The thermal mass of the fixture inside the bath should be minimized so that the fixture and the bath are uniform.

X1.7 Sample size is limited to diameter or thickness in the range of 0.3 to 3.0 mm. Larger samples can be tested with this test method with the addition of a fixture to aid in the deformation of the sample. Smaller samples cannot be easily tested with this test method because of friction in the motion-recording system. Smaller samples, however, can be tested using a similar approach in which the motion is recorded with a vision system.