Designation: F2082 – 06

# StandardTest 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; 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 either fully-annealed or heat-treated 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:*[2]

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

F2005 Terminology for Nickel-Titanium Shape Memory Alloys

## 3. Terminology

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

3.2 *free recovery*—unconstrained motion of a shape memory alloy upon heating and transformation to austenite after deformation in a lower temperature phase.

3.3 *Abbreviations:*

3.3.1 *LVDT*—linear variable differential transducer.

3.3.2 *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 F2005, 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

[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.

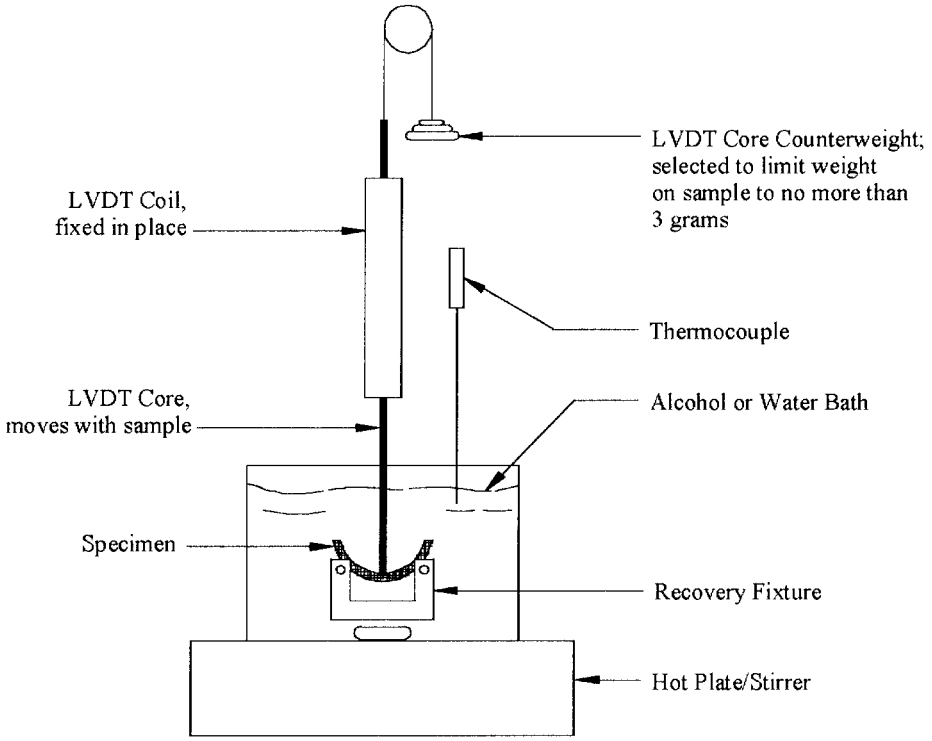


**FIG. 1 Schematic Showing Side View of Test Apparatus Using a Vertically Mounted and Counterbalanced LVDT (LVDT Power Supply, Thermocouple Indicator, and Data Acquisition System Are Not Shown)**

greater than 45°, with power supply, mounted in an appropriate fixture (see Fig. 2); or vision system; or equivalent means of measuring sample displacement.

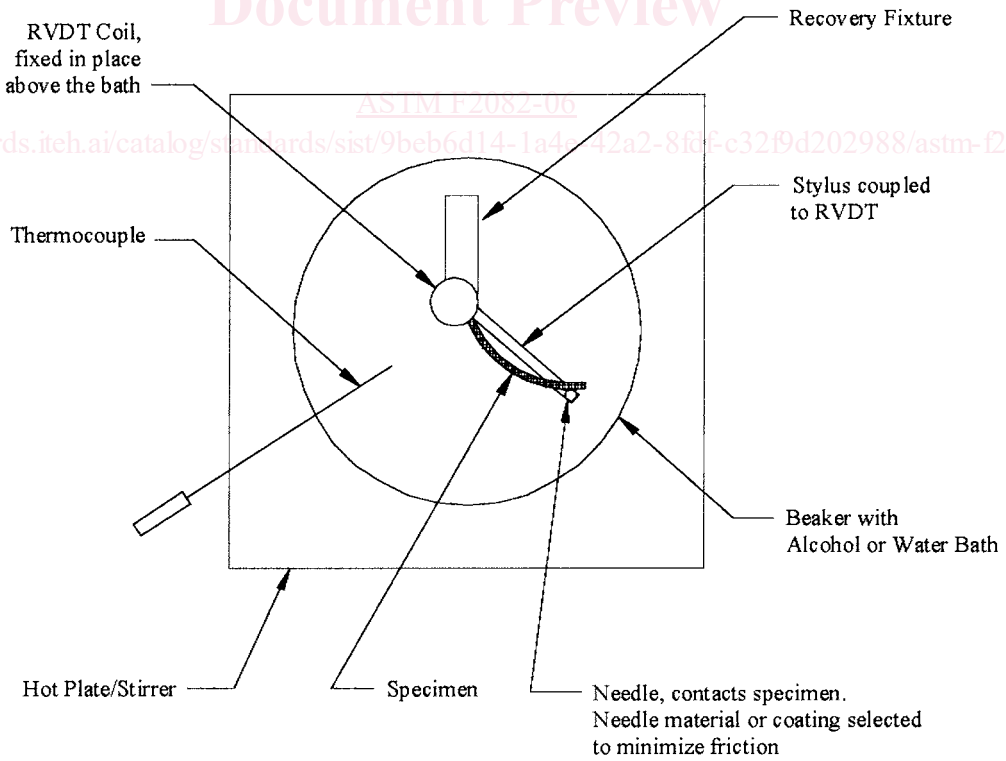


**FIG. 2 Schematic Showing Top View of Test Apparatus Using an RVDT (RVDT Power Supply, Thermocouple Indicator, and Data Acquisition System Are Not Shown)**

6.2 *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 *Bath of Heat Transfer Fluid,* for example, denatured alcohol, ethylene glycol, water, and so forth.

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 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 diameter or thickness may be less than 0.3 mm.

7.2 Specimens may be tested in the semifinished (heat-treated) or annealed condition. Anneal is defined in Terminology F2005.

## 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 at room temperature, cool a bath of appropriate heat transfer fluid to –55°C (–67°F) or lower using liquid nitrogen, dry ice, or other suitable method. For alloys that are martensitic at room temperature, cool the 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 2.5 %. For these strains, mandrel diameter shall be between 39 and 49 times specimen diameter or thickness.

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 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 must be such that the temperature of the fixture and the bath is uniform throughout the test.

9.8 Set the device 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 must be counterbalanced such that the weight on the specimen is no more than 3 g.

9.8.2 For an RVDT, 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 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 $A_f$. 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. Determination of Transformation Temperature

10.1 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



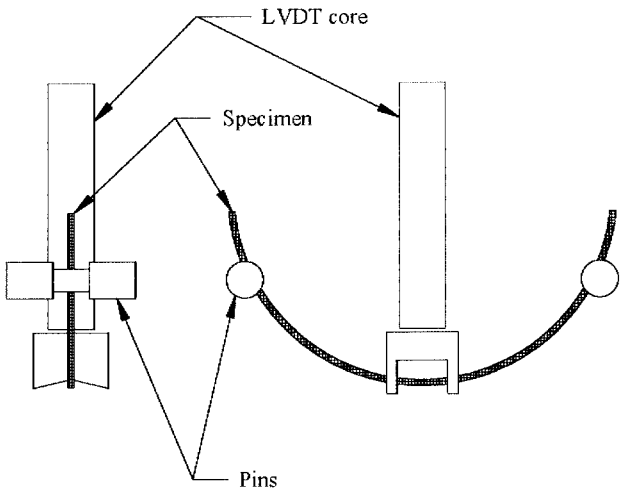


**FIG. 3 Placement of LVDT Core on Deformed Specimen, Which Is Resting on Recovery Fixture Pins**