



Designation: F 2082 – 03

## Standard Test Method for Determination of Transformation Temperature of Nickel- Titanium Shape Memory Alloys by Bend and Free Recovery<sup>1</sup>

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 ( $\epsilon$ ) 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<sup>2</sup>

F 2005 Terminology for Nickel-Titanium Shape Memory Alloys<sup>3</sup>

### 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 transforma-

tion, 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  $+90^\circ\text{C}$ .

### 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  $45^\circ$ , with power supply, mounted in an appropriate fixture (see Fig. 2).

6.2 *Thermocouple and Indicator*, with resolution of  $0.1^\circ\text{C}$  ( $0.2^\circ\text{F}$ ) 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.

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 13.01.

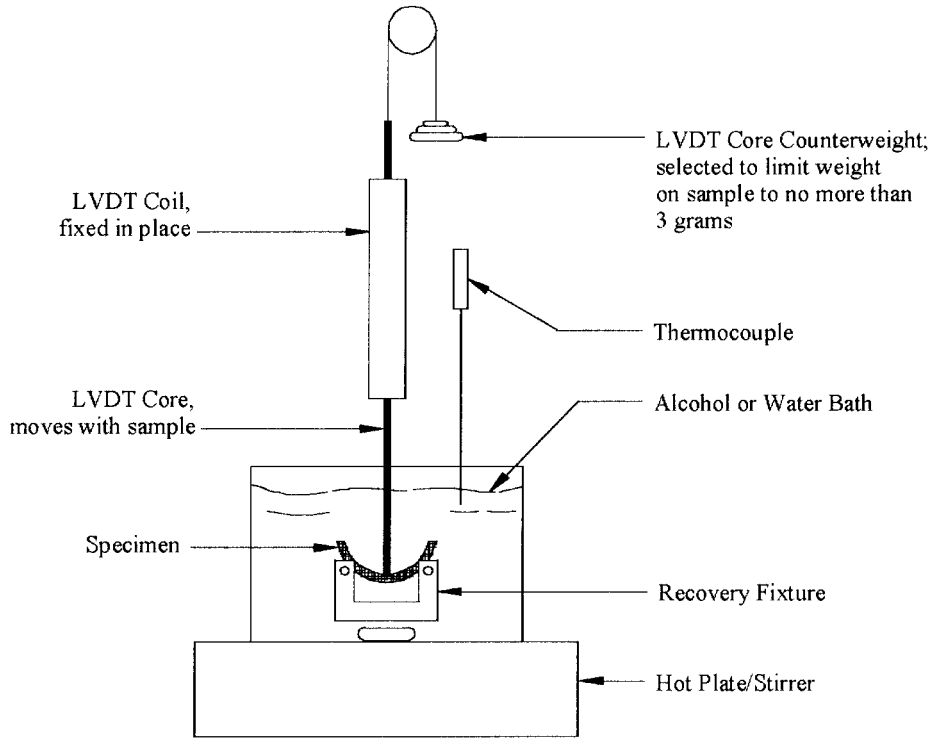


FIG. 1 Schematic Showing Side View of Test Apparatus Using a Vertically Mounted and Counterbalanced LVDT (LVDT Power Supply, Thermocouple Indicator, and Data Acquisition System Are Not Shown)

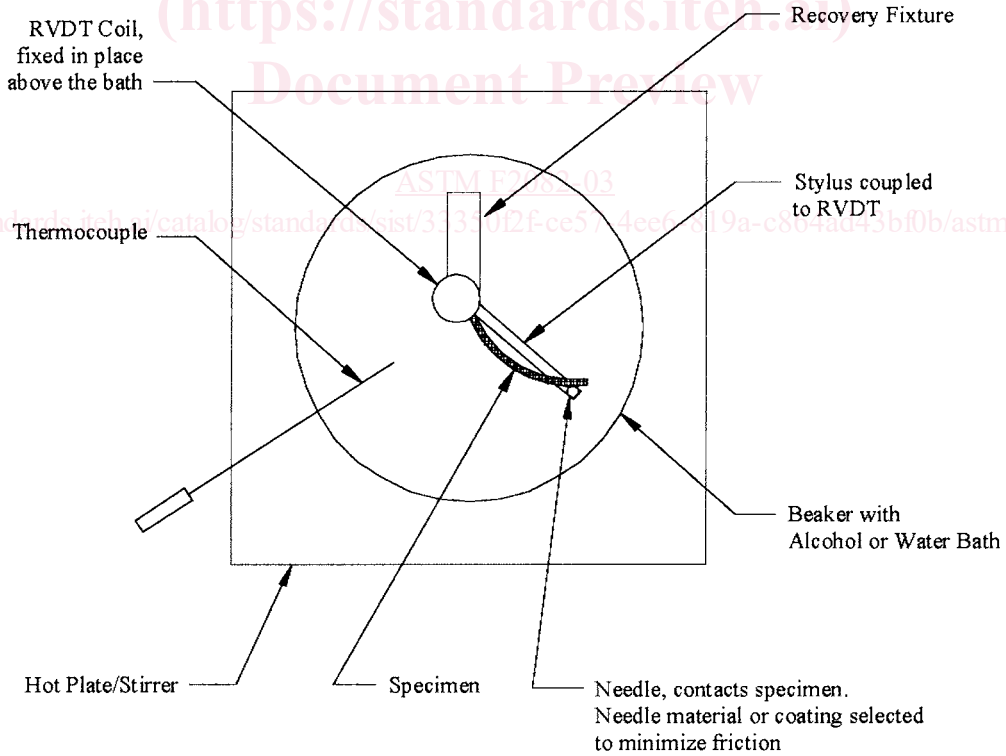


FIG. 2 Schematic Showing Top View of Test Apparatus Using an RVDT (RVDT Power Supply, Thermocouple Indicator, and Data Acquisition System Are Not Shown)

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  $-55^{\circ}\text{C}$  ( $-67^{\circ}\text{F}$ ) or lower using liquid nitrogen or dry ice. For alloys that are martensitic at room temperature, cool the water bath to  $10^{\circ}\text{C}$  ( $50^{\circ}\text{F}$ ) 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  $180^{\circ}$  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  $180^{\circ}$  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.

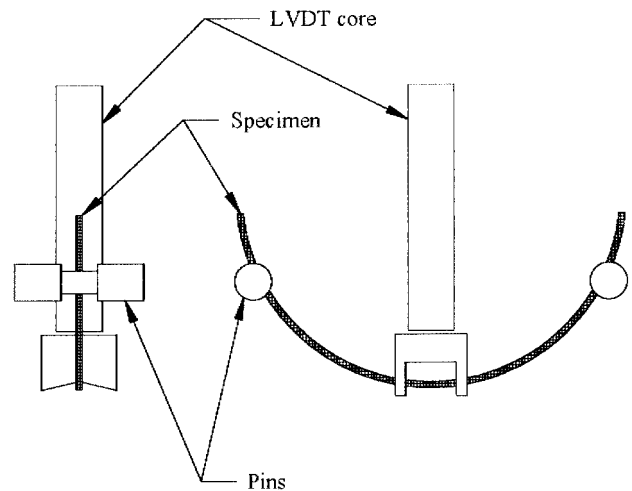
9.11 Stir and heat the bath on the hot plate to a temperature above the  $A_f$ . Limit the heating rate to no more than  $4^{\circ}\text{C}/\text{min}$  during the recovery.

9.12 Stop the test once the temperature is at least  $10^{\circ}\text{C}$  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  $-55^{\circ}\text{C}$  ( $-67^{\circ}\text{F}$ ) or lower using liquid nitrogen or dry ice. For alloys that are martensitic at room temperature, cool the water bath to  $10^{\circ}\text{C}$  ( $50^{\circ}\text{F}$ ) or lower.

10.2 Select a mandrel according to the sample diameter or thickness to give a strain of 2 to 2.5 %. For these strains,



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

mandrel diameter shall be between 39 and 49 times specimen diameter or thickness.

10.3 Cut a test specimen long enough to wrap  $90^{\circ}$  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 3 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  $90^{\circ}$  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  $4^{\circ}\text{C}/\text{min}$  during the recovery.

10.12 Stop the test once the temperature is at least  $10^{\circ}\text{C}$  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.