Designation: F2082/F2082M – 16

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 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 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 with 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]

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

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

[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.

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 the 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); 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) 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)

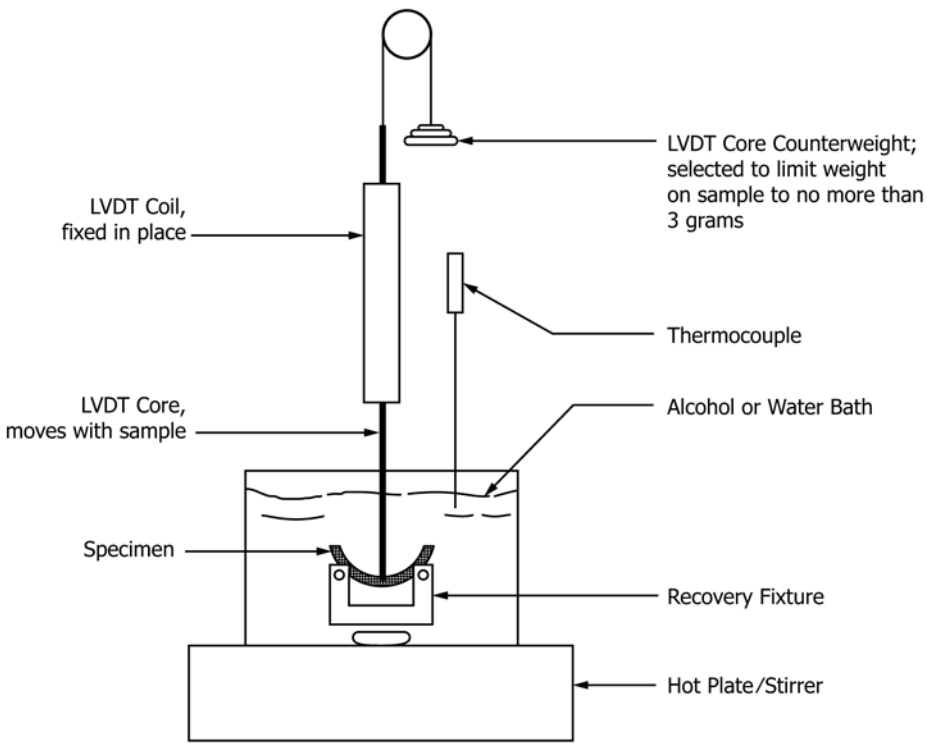

FIG. 1 Schematic Showing Side View of Test Apparatus Using a Vertically Mounted and Counterbalanced LVDT (LVDT Power Supply, Thermocouple Indicator, and Data Acquisition System are not shown)

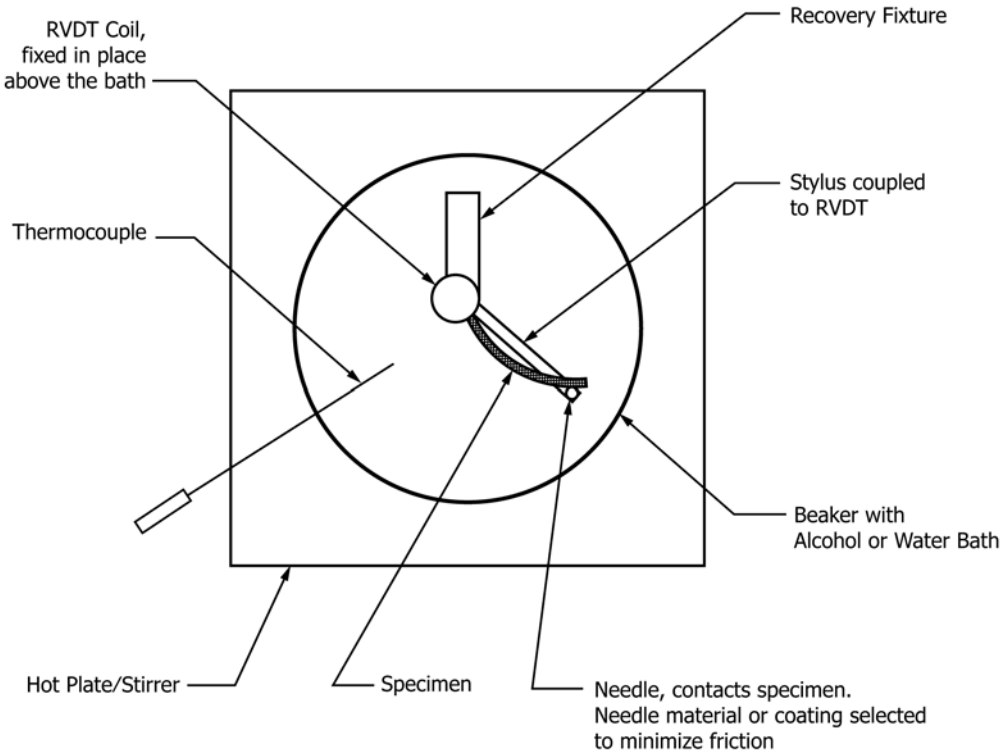

FIG. 2 Schematic Showing Top View of Test Apparatus using an RVDT (RVDT Power Supply, Thermocouple Indicator, and Data Acquisition System are not shown)

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 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.

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 during the recovery.

9.12 Stop the test once the temperature is at least 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'_s, 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 and Fig. 6. The transformation may occur in one or two stages. For a one-stage transformation, the middle tangent line should be

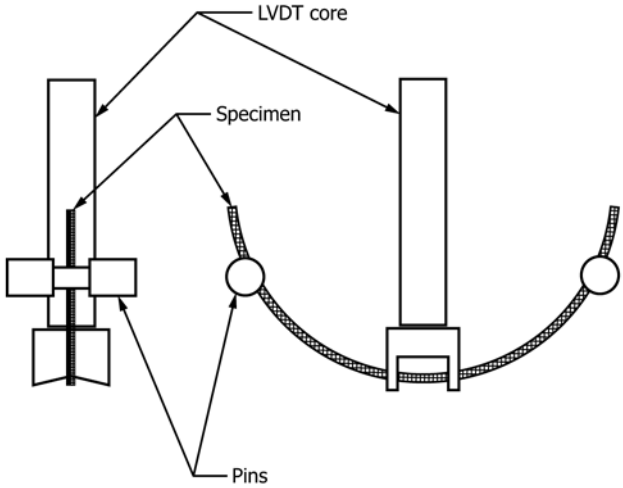

FIG. 3 Placement of LVDT Core on Deformed Specimen, which is resting on Recovery Fixture Pins

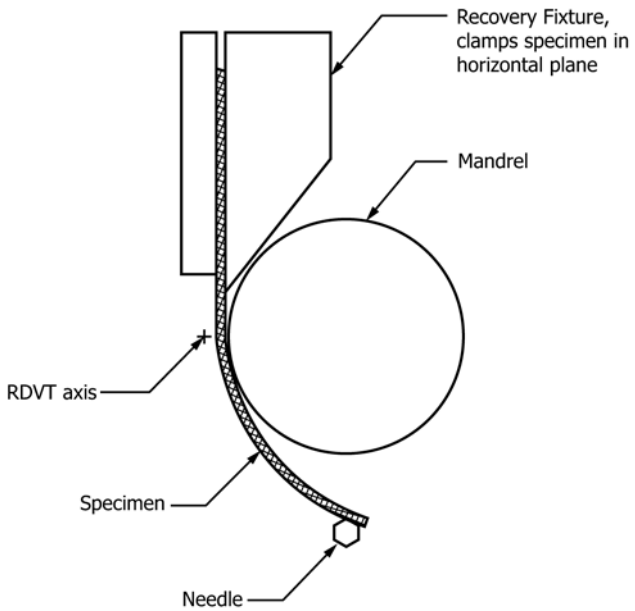

FIG. 4 Placement of Needle on Deformed Specimen, which is Clamped to the Recovery Fixture. Top View Shown with Stylus and RVDT Removed. Note that the Recommended RVDT Axis Location is Offset from the Mandrel by the Radius of the Needle

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., 0 % recovery). A 100 % recovery of deformation shall be considered to occur 10°C beyond the temperature that the displacement-versus-temperature curve attains a constant slope.

11. Report

11.1 The report shall include the following information:

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

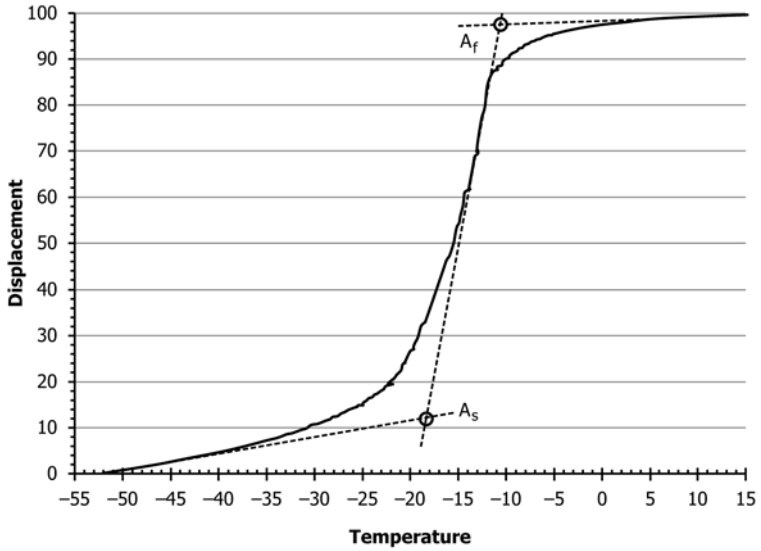

FIG. 5 Tangent Method: One-Stage Transformation—Tangent Lines and Transformation Temperatures

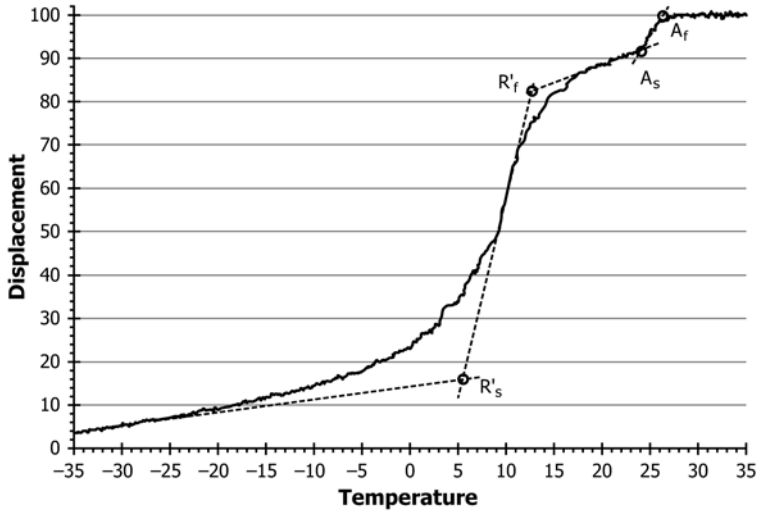

FIG. 6 Tangent Method: Two-Stage Transformation—Tangent Lines and Transformation Temperatures

TABLE 1 Repeatability and Reproducibility for A_s of Cold-Worked and Stress-Relieved Material

Material	A_s, grand mean	Repeatability standard deviation	Reproducibility standard deviation	Repeatability	Reproducibility
A	–29.0	1.4	3.4	4.1	9.5
B	–12.4	2.5	4.5	6.9	12.5

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

12. Precision and Bias

12.1 An interlaboratory study was conducted in accordance with Practice E691 in six laboratories with two different materials, with each laboratory obtaining five results for each material. There were two rounds of testing. In the first round, the test samples were cold-worked and stress-relieved; in the

TABLE 2 Repeatability Limit and Reproducibility Limit for $A_{f\text{-}tan}$ of Cold-Worked and Stress-Relieved Material

Material	A_f, grand mean	Repeatability standard deviation	Reproducibility standard deviation	Repeatability	Reproducibility
A	–11.7	1.8	4.5	4.9	12.7
B	2.9	1.5	4.8	4.3	13.6

TABLE 3 Repeatability Limit and Reproducibility Limit for A_s of Annealed Material

Material	A_s, grand mean	Repeatability standard deviation	Reproducibility standard deviation	Repeatability	Reproducibility
A	–25.4	1.4	2.2	4.1	6.3
B	0.2	1.9	2.7	5.3	7.7

TABLE 4 Repeatability Limit and Reproducibility Limit for $A_{f\text{-}tan}$ of Annealed Material

Material	A_f, grand mean	Repeatability standard deviation	Reproducibility standard deviation	Repeatability	Reproducibility
A	–18.6	1.7	2.4	4.6	6.8
B	8.3	0.9	1.7	2.7	4.8

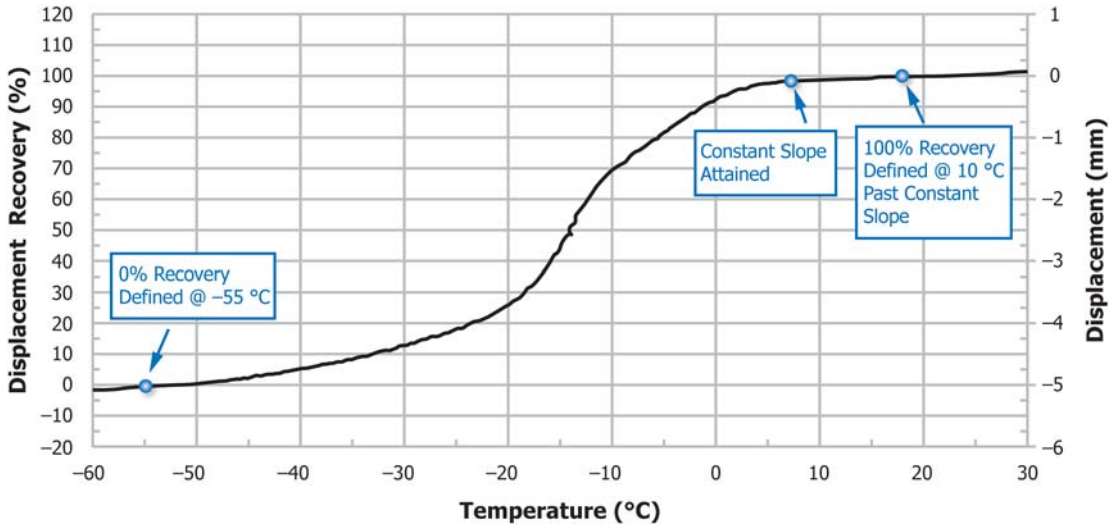

FIG. 7 Percent Recovery Method: $A_{f\text{-}95}$ Transformation Temperature

second round, the samples were fully annealed. The details are given in Research Report No. F04–1009.[3]

12.2 The results of round one are shown in Table 1 and Table 2 for each transformation temperature (A_s, $A_{f\text{-}tan}$). The values are in degrees Celsius. The terms repeatability limit and reproducibility limit are used as specified in Practice E177.

12.3 The results of round two are shown in Table 3 and Table 4 for each transformation temperature (A_s, $A_{f\text{-}tan}$). The values are in degrees Celsius. The terms repeatability limit and reproducibility limit are used as specified in Practice E177.

12.4 *Bias*—No measurement of bias is possible with this test method because there is no accepted reference material or method.

13. Keywords

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

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F04-1009.

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, economical means of determining the martensitic-to-austenitic transformation temperatures by recording the motion of the samples as they exhibit 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 2.5 % 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 in temperature.

X1.7 Samples larger than 3 mm in thickness or diameter can be tested with this test method with the addition of a fixture to aid in the deformation of the sample. A vision system is recommended for samples less than 0.3 mm, because friction in an RVDT or LVDT system may create a stress in the specimen.

X1.8 Material with retained cold work may exhibit a "sluggish" recovery, similar to that shown in Fig. 7. For such cases, the percent recovery method may be preferred to the tangent method, because the determined A_f can be closer to the complete martensitic-to-austenitic transformation. Determining the temperature at which the material is fully recovered is not practical because the last 1 to 2 % of the motion is typically spread over several degrees, leading to variability in determining when to stop the test and determine a point of 100 % recovery.

X1.9 The $A_{f\text{-}95}$ method was added to this standard in 2016. Documents referencing A_f by bend free recovery are to be interpreted to mean A_f by the tangent method ($A_{f\text{-}tan}$).