Designation: F2004 – 24

Standard Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis[1]

This standard is issued under the fixed designation F2004; 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 defines procedures for quantitatively determining the transformation temperatures and enthalpies of nickel-titanium shape memory alloys, produced in accordance with Specification F2063, by differential scanning calorimetry.

1.2 This test method may be used for heat-treated samples in the aged, annealed shape-set, or tempered condition. See Terminology F2005 for definitions of these heat treat conditions.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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 to determine the applicability of regulatory limitations prior to use.*

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E473 Terminology Relating to Thermal Analysis and Rheology

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E967 Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers

E1142 Terminology Relating to Thermophysical Properties

F2005 Terminology for Nickel-Titanium Shape Memory Alloys

F2063 Specification for Wrought Nickel-Titanium Shape Memory Alloys for Medical Devices and Surgical Implants

F2082 Test Method for Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery

3. Terminology

3.1 Specific technical terms used in this test method are found in Terminologies E473, E1142, and F2005.

4. Summary of Test Method

4.1 This test method involves heating and cooling a test specimen at a controlled rate in a controlled environment through the temperature interval of the phase transformation. The difference in heat flow between the test material and a reference material due to energy changes is continuously monitored and recorded. Absorption of energy due to a phase transformation in the specimen results in an endothermic peak on heating. Release of energy due to a phase transformation in the specimen results in an exothermic peak on cooling.

5. Significance and Use

5.1 Differential scanning calorimetry provides a rapid method for determining the transformation temperature(s) and the enthalpies of transformation of nickel-titanium shape memory alloys.

5.2 This test method uses small, stress-free, annealed samples to determine whether a sample of nickel-titanium alloy containing nominally 54.5 to 57.0 % nickel by weight is austenitic or martensitic at a particular temperature. Since chemical analysis of these alloys does not have sufficient precision to determine the transformation temperature by measuring the nickel-to-titanium ratio of the alloy, direct

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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.

measurement of the transformation temperature of an annealed sample of known thermal history is recommended.

5.3 This test method is useful for quality control, specification acceptance, and research.

5.4 Transformation temperatures derived from differential scanning calorimetry (DSC) may not agree with those obtained by other test methods due to the effects of strain and load on the transformation. For example, transformation temperatures measured in accordance with Test Method F2082 will differ from those measured by this standard.

5.5 The use of this test method for finished or semi-finished components in the aged, shape-set, or tempered conditions shall be agreed upon between the purchaser and the supplier. See Terminology F2005 for the definitions of these heat treat conditions.

6. Interferences

6.1 Make sure the material to be tested is homogeneous since milligram sample quantities are used.

6.2 Take care in preparing the sample (**1**). Cutting and grinding can cause localized heating and/or deformation that affect the transformation temperature. Oxidation during heat treatment can change the thermal conductance of the sample.

NOTE 1—Cutting with an abrasive saw with coolant, such as a low-speed diamond saw, has been found to work well for sectioning samples into segments suitable for the (DSC) sample pan. The abrasive cutting action minimizes cold work while the coolant minimizes localized heating.

NOTE 2—The recommended operating conditions for low-speed diamond saw cutting are blade edge rotation of 89 to 109 meters per minute with coolant at 20 to 23 °C.

6.3 Set the gas flow to provide adequate thermal conductivity in the test cell.

7. Apparatus

7.1 Use a differential scanning calorimeter capable of heating and cooling at rates up to 10 °C/min and of automatically recording the differential energy input between the specimen and the reference to the required sensitivity and precision.

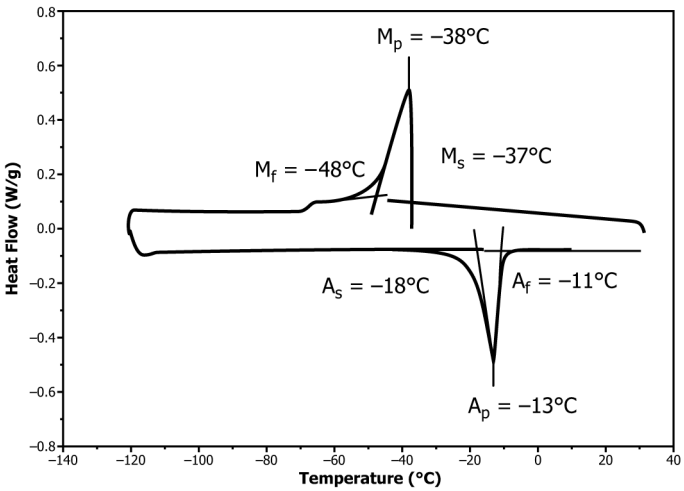

FIG. 1 DSC Curve for Nickel-Titanium (NiTi)

7.2 Confirm that the sample weight is large enough for reliable operation of the calorimeter.

7.3 Use sample capsules or pans composed of aluminum or other inert material of high thermal conductivity.

7.4 Use helium gas purge supply. See 10.3.1.

7.5 Use an analytical balance with a capacity of 100 mg capable of weighing to the nearest 0.1 mg.

8. Sampling

8.1 Sample weight will affect TTR (transformation temperature range, F2005) particularly A_f and M_f. See Ref (**1**), Figure 6. When comparing test results, make sure that the sample weights are similar so as not to affect the results.

8.1.1 Use a sample weight based on wire, tube, or strip diameter or thickness based on the guidance shown in Table 1. Cut the sample to maximize surface contact with the calorimeter sample pan.

8.1.2 The minimum sample weight to obtain a thermogram sufficient for analysis will depend on the enthalpy of the transformation reaction, the heating and cooling rate, and the sensitivity of the calorimeter. Make sure the sample weight is large enough to yield adequate endothermic and exothermic energy peaks compared to the baseline noise of the calorimeter.

8.1.3 Sample weights outside the ranges shown in Table 1 may be used if the amount of material available for sampling is limited and the calorimeter is able to yield an adequate signal as described in 8.1.2. The range of sample weights in Table 1 shall be considered requirements if the testing is done for quality control or specification acceptance.

8.2 Annealed samples shall be heat treated at 800 to 850 °C for 15 to 60 min in vacuum or inert atmosphere, or in air with adequate protection from oxidation. Rapidly cool the sample to prevent precipitation of phases that may change the transformation temperature of the alloy. For example, bare 10 to 45 mg samples can be air cooled after heat treating.

8.3 Samples of device components shall be tested as received if they have already been aged, shape-set, or tempered as required for the application.

8.4 Clean the sample of all foreign materials such as cutting fluid. If the sample is oxidized during heat treatment, grind, polish, or etch the sample to remove the oxide. Take care to avoid cold working the sample as this will change its thermal response. Slight oxidation is permissible, but remove all heavy oxide scale.

9. Calibration

9.1 Calibrate the temperature axis of the instrument using the same heating rate, purge gas, and flow rate as those used for analyzing the specimen in accordance with Test Method E967.

TABLE 1 Sample Weight for Product Dimensions

Wire, Tube, or Strip Diameter or Thickness	Sample Weight, mg
<0.305 mm (0.012 in.)	10–45
≥0.305 mm (0.012 in.)	25–45