Designation: F1978 – 22

Standard Test Method for Measuring Abrasion Resistance of Metallic Thermal Spray Coatings by Using the Taber Abraser[1]

This standard is issued under the fixed designation F1978; 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 quantifies the abrasion resistance of metallic coatings produced by thermal spray processes on flat metallic surfaces. It is intended as a means of characterizing coatings used on surgical implants.

1.2 This test uses the Taber Abraser,[2] which generates a combination of rolling and rubbing to cause wear to the coating surface. Wear is quantified as cumulative weight loss.

1.3 This test method is limited to flat, rigid specimens that do not react significantly with water and do not undergo a phase transformation or chemical reaction between room temperature and 100 °C in air.

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

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

1.6 *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:*[3]

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

G195 Guide for Conducting Wear Tests Using a Rotary Platform Abraser

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *abraser, n*—instrument that is designed to determine the resistance of surfaces to composite rolling and rubbing action.

3.1.2 *particle shedding, n*—loss of surface particles and fragments from a coating.

3.1.3 *thermal spray coating, n*—coating produced by spraying melted or softened powder or wire by means of combustible gases, plasma, or two-wire arc.

3.1.4 *weight loss, n*—amount of mass removed by the test apparatus over the course of testing.

4. Summary of Test Method

4.1 This test method uses a Taber Abraser with H-22 Calibrade (trademarked) wheels[2] and the 250 g mass of the abrading head without added weights. A specimen is abraded using rotary rubbing action under controlled conditions of pressure and abrasive action. The test specimen, mounted on a turntable platform, turns on a vertical axis against the sliding rotation of two abrading wheels. The wheels shall be mounted in such a way that when they are in contact with the rotating test specimen, they rotate in opposing directions. One abrading wheel rubs the specimen outward toward the periphery and the other inward toward the center while a vacuum system removes wear debris during the test. The resulting abrasion

[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] Trademarked. The sole source of supply of the apparatus known to the committee at this time is Taber Industries, North Tonawanda, NY 14120 USA. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[3] 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.

*A Summary of Changes section appears at the end of this standard

marks form a pattern of crossed arcs over an area of approximately 30 cm^2. Specimens are abraded, cleaned ultrasonically, dried, cooled, and weighed for a set number (2, 5, 10, and 100) of cumulative rotational cycles. The specimens are weighed after each iteration, and the weight loss is the measure of abrasive wear to the specimen.

5. Significance and Use

5.1 This test method provides a means to evaluate the resistance to particle shedding of a thermal spray coating. Such particle shedding might occur during surgical insertion of an implant or as the result of micromotion of the implant after insertion.

5.2 This abrasion test method may be useful for quality control analysis of a coating, and it can be used to evaluate the effects of processing variables, such as substrate preparation before coating, surface texture, coating technique variables, or postcoating treatments, any of which may influence the susceptibility of the coating to particle shedding.

5.3 This abrasion test method is for flat plate-shaped specimens of a size sufficient that the wheels of the abrader do not leave the surface of the specimen. It is not recommended for devices with other shapes or sizes.

6. Apparatus

6.1 *Taber Abraser* (Fig. 1), or equivalent as described in Guide G195, with abrading head of 250 g mass and no added weights, and consisting of the following elements:

6.1.1 A specimen turntable platform, which is removable, that includes a rubber pad and centrally located threaded post and nut.

6.1.2 A motor capable of rotating the turntable platform at a speed of either 72 r/min ± 2 r/min or 60 r/min ± 2 r/min.

6.1.3 A pair of abrading heads, which include the pivoted arm and flanged holder to which the abrasive wheel is attached.

6.1.4 A vacuum suction system and vacuum pickup nozzle to remove debris and abrasive particles from the specimen surface during testing. A vacuum suction level of 100 shall be used and the vacuum suction force shall be 13.7 kPa or greater, as measured by a vacuum gauge at the vacuum pickup nozzle port. The height of the vacuum pickup nozzle shall be adjustable.

6.1.5 A counter to record the number of abrasion cycles (revolutions) made by the turntable platform.

6.2 *H-22 Taber Calibrade Wheels,*[2] vitrified based, consisting of hard particles embedded in a binder material.

6.2.1 The wheels shall be cylindrically shaped; 12.7 mm ± 0.3 mm thick; include an axial hole 16.0 mm ± 0.1 mm to allow the wheel to be mounted to the flanged holder on the pivoted arm; and have an external diameter of 51.9 mm ± 0.5 mm when new, and in no case less than 44.4 mm.

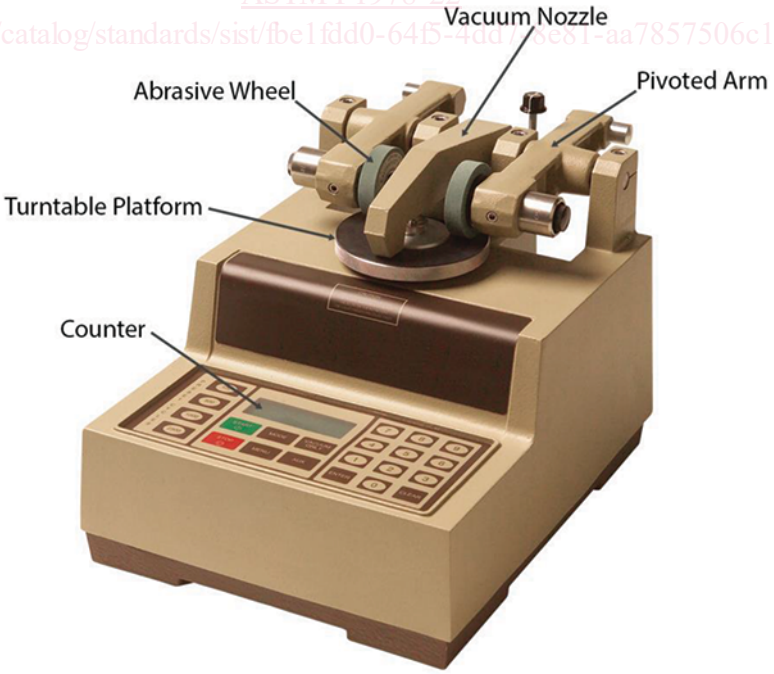
6.3 *Taber Wheel Refacer,*[2] or equivalent.

6.4 *Ultrasonic Cleaning Unit,* for cleaning specimens after abrading.

6.5 *Drying Oven,* capable of operation at 100 °C ± 2 °C, for drying specimens.

6.6 *Analytical Balance,* capable of weighing specimens with a resolution of 0.0001 g.

6.7 *Infrared Thermometer* (optional), to measure temperature of specimens before weighing.



NOTE 1—Vacuum suction system not shown.

FIG. 1 Taber Abraser

6.8 *Deionized Water.*

6.9 *Sodium Chloride (NaCl),* reagent grade.

7. Test Specimen

7.1 Abrasion test specimens shall be approximately 10 cm squares or 10 cm diameter circles of at least 1.6 mm but not greater than 6.5 mm thickness, with a 6.5 mm diameter hole through the center to allow the specimen to be secured to the specimen holder of the Taber Abraser. For substrates other than titanium, consideration shall be given to the weight of the test specimen relative to the capacity of the analytical balance.

7.2 The coating shall be applied in a manner representative of that used on the finished surgical implant.

7.3 Prepare a minimum of seven specimens. If there is more than one coating to evaluate, a minimum of six samples for each coating shall be prepared and at least one set shall contain seven samples.

7.3.1 Of the seven-sample set, one sample shall be selected to determine the time required for ultrasonic cleaning.

7.3.2 One specimen, randomly selected from each group of six specimens, shall be reserved to measure weight loss caused by ultrasonic cleaning. This specimen shall be called the "blank" specimen and shall be weighed, ultrasonically cleaned, dried, cooled, and reweighed for an equal number of times of the abraded specimen.

8. Procedure

8.1 *General Information:*

8.1.1 If there is more than one set of specimens to test, the specimens shall be tested in a random sequence.

8.1.2 A single complete specimen run shall consist of a series of partial runs, commencing with an initial two-cycle partial run and continuing until 100 cycles have been completed. Once a specimen run is initiated, no other specimens shall be tested using the same wheel until the specimen run is finished.

8.1.3 At the start of each new complete specimen run, the display indicating the number of cycles run shall be reset. The Taber Abraser counts cumulative cycles (cycles completed) and so the number of cycles set for each partial run shall be the cumulative number of cycles (2, 5, 10, or 100) designated as the end of that partial run. See Note 4.

8.2 *Determining Time of Ultrasonic Cleaning*—Before running the battery of tests, the time required for ultrasonic cleaning shall be determined.

8.2.1 Using the analytical balance, weigh the seventh sample as described in 7.3.1 no fewer than three times and record the average weight of these measurements.

8.2.2 Prepare the Taber Abraser following steps 8.4.2 – 8.4.4.

8.2.3 Set the Taber Abraser for ten cumulative cycles and start the turntable.

8.2.4 Clean the sample for 10 min with a fresh saline solution (see 8.4.1), with the ultrasonic cleaner to be used for the coating analysis.

NOTE 1—To ensure wear debris is effectively removed, the specimen side subjected to abrasion should be facing downward during ultrasonic cleaning. Utilize a support that will prevent the specimen from contacting the bottom of the ultrasonic cleaning unit.

8.2.5 Place the cleaned sample in a 100 °C oven and dry for a minimum of 10 min.

8.2.6 Allow the sample to cool to within 1 °C of room temperature before weighing.

NOTE 2—Specimens that are not cooled sufficiently may generate convection currents and result in erroneous mass readings. An infrared thermometer can be used to measure the temperature of the specimen prior to weighing.

8.2.7 Using the analytical balance, weigh the sample no fewer than three times and record the average weight of these measurements.

8.2.8 Repeat 8.2.4 – 8.2.7 until the same mass (within ±0.0003 g) is recorded for two consecutive cleanings.

8.2.9 Record the total number of cleanings performed in 8.2.4 – 8.2.8.

8.2.10 Determine the required cleaning time necessary to obtain a stable mass, as $(10 \cdot (x - 1))$ min, where x is the total number of cleanings determined in 8.2.9.

NOTE 3—Handle specimens by the edges to prevent unintentional particle shedding.

8.3 *Reference Standard (Blank)*—Each group of six specimens shall include one unabraded "blank" specimen, as specified in 7.3.2. Once in every six tests, an unabraded specimen shall be used as a control to determine the weight loss caused by the ultrasonic cleaning. This specimen shall be weighed, cleaned for the time calculated in 8.2.10, dried, cooled, and reweighed for as many times as would normally be done if abrasion testing were being performed. These values shall be logged as "blank" weight loss values.

8.4 *Operation:*

8.4.1 Prepare a fresh cleaning solution for the ultrasonic cleaner by adding 0.1 g ± 0.005 g of reagent grade NaCl to each litre of deionized water.

8.4.2 Resurface the abrading wheels using the Taber Wheel Refacer for each new specimen. Check the diameter of the wheel. It is essential that the diameter of the abrading wheels not fall below the marked minimum level (44.4 mm) over the course of the test. Should the wheel diameter fall below the labeled mark, the test run shall be discarded and a new specimen shall be run in its place with a new pair of abrading wheels.

8.4.3 Mount the H-22 Calibrade wheels by placing the wheel marked "Left Wheel" on the left-hand abrading head and the wheel marked "Right Wheel" on the right-hand abrading head, with the labels facing toward each other.

8.4.4 Abrasion shall be done with only the 250 g mass of the abrading head assembly. No extra weights shall be added to the abrading head.

8.4.5 Clean the specimen for the time determined in 8.2.10 using an ultrasonic cleaner with saline solution as defined in 8.4.1. This solution shall be changed to a fresh solution before the first cleaning done that day. See Note 1.

8.4.6 Place the cleaned specimen in a 100 °C oven and dry for a minimum of 10 min.

8.4.7 Allow the specimen to cool to within 1 °C of room temperature before weighing. See Note 2.

8.4.8 Using the analytical balance, weigh the specimen no fewer than three times, and record the average of these weight measurements on a data sheet.

8.4.9 Place the specimen on the Taber Abraser turntable, coating side up, such that the screw projects through the hole in the center of the specimen. Secure the specimen to the turntable using the nut.

8.4.10 Lower the vacuum pickup nozzle. The gap between the specimen and vacuum pickup nozzle shall be adjusted to 3 mm.

8.4.11 Lower the abrading heads gently so that the abrading wheels rest on the surface of the specimen.

8.4.12 Set the Taber Abraser to reach the appropriate number of cumulative cycles (2, 5, 10, or 100). Start the Taber vacuum prior to beginning the test and let the vacuum run for no less than 5 s to allow the vacuum to reach full power, then start the test.

NOTE 4—The initial partial run shall be two cycles. The second partial run shall be three cycles (five cumulative cycles). The third partial run shall be five cycles (ten cumulative cycles). And the fourth partial run shall be 90 cycles (100 cumulative cycles).

8.4.13 Clean, dry, cool, and weigh the specimen as before (see 8.4.5 – 8.4.8).

8.4.14 Repeat 8.4.9 – 8.4.13 for 5, 10, and 100 cumulative cycles.

8.4.15 Repeat the steps specified in 8.4.2 – 8.4.14 for all specimens not reserved as blanks for the test.

8.4.16 Run each blank through the steps specified in 8.4.5 – 8.4.14, skipping the steps specified in 8.4.9 – 8.4.12.

9. Calculation

9.1 Calculate the cumulative weight loss for each specimen for the number of revolutions used with the following equation:

$$\Delta w_n = \langle w_o \rangle - \langle w_n \rangle \quad (1)$$

where:

n = number of cumulative cycles to which specimen has been exposed (2, 5, 10, 100),
Δw_n = cumulative weight loss for n cycles,
$\langle w_o \rangle$ = average of three weight measurements at the start of the test, and
$\langle w_n \rangle$ = average of three weight measurements after n cumulative cycles.

10. Report

10.1 The report shall include the following information:

10.1.1 Identification of the test coupon materials, including traceability information such as coating process lot and raw material lot numbers.

10.1.2 Dimensional data including coupon dimensions and coating thickness dimensions.

10.1.3 The number of specimens tested.

10.1.4 Nominal laboratory temperature and humidity during the test.

10.1.5 The number of minutes needed for ultrasonic cleaning, as determined in 8.2.10.

10.1.6 All cumulative mass losses following abrasion and/or ultrasonic cleaning associated with each specimen or blank.

10.1.7 The mean cumulative mass loss and its standard deviation for 2, 5, 10, and 100 cycles.

10.1.8 Observations made during the course of the study including, but not limited to: observation of wear tracks and when they occur, the appearance of discoloration of the specimen, the removal of large sections of coating, and evidence of corrosion.

11. Precision and Bias

11.1 An interlaboratory study (ILS) was conducted to establish the precision and bias of this test method. The number of laboratories, materials, and determinations in this study meet the minimum requirements for determining precision as prescribed in Practice E691 (see Table 1). These tests were done for 2, 5, 10, and 100 cycles for a total of twelve material and cycle combinations.

11.2 *Precision*—Precision, characterized by repeatability, S_r, and reproducibility, S_R, have been determined for the materials and cycle combinations provided in Table 2.

12. Keywords

12.1 abrasion; particle shedding; porous coatings; Taber Abraser; thermal spray coatings; wear

TABLE 1 ILS Study Parameters

	This Study	Practice E691 min
Laboratories	6	6
Materials	3	3
Determinations	5	2