Designation: F 1978 – 00ε1

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 F 1978; 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.

ε[1] NOTE—Tables 1 and 2 were editorially corrected in May 2001.

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™[2] abraser, which generates a combination of rolling and rubbing to cause wear to the coating surface. Wear is quantified as cumulative mass 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 *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 applicable regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

3.1.2 *particle shedding, n*—the 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™ wheels[2] and the 250-g mass of the abrading head without added weights. Specimens are abraded repeatedly and cleaned ultrasonically for a set number of rotational cycles (2 to 100 cycles). The specimens are weighed after each cleaning, and the mass 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 sprayed coating. Such particle shedding might occur during surgical insertion of an implant or as the result of micromotion of the implant after surgical 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, however, for devices with other shapes or sizes.

6. Apparatus

6.1 *Taber™ Abraser Model 5150*, or equivalent.

6.2 *Two H-22 Taber™ Calibrade™ Wheels*, or equivalent, with abrading head of 250-g mass and no added weights.

6.3 *Taber™ Vacuum Unit*, made by Shop-Vac™,[2] 7.4 amps, or equivalent.

6.4 *Taber™ Wheel Refacer Model 200*[2].

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

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

6.7 *Analytical Balance*, capable of weighing specimens to

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[2] 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, which you may attend.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

an accuracy of 0.0001 g.

7. Test Specimen

7.1 Abrasion test specimens shall be 10-cm (4-in.) squares or 10-cm diameter circles of at least 0.16-cm (0.0625-in.) thickness with a 0.64-cm (0.25-in.) diameter hole through the center to allow the specimen to fit on the pin 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 A minimum of six samples for each coating shall be tested. At least one set shall contain seven samples.

7.4 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, cleaned, and re-weighed for an equal number of times to the cleaning and weighing of the abraded specimen.

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

8. Procedure

8.1 *General Test Conditions*:

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

8.1.2 Resurface the abrading wheels using Taber™ Wheel Refacer Model 200 for each new specimen. Check the radius of the wheel, it is essential that the radius of the abrading wheels not fall below the marked minimum level over the course of the test. Should the wheel radius fall below the labeled mark, the test run shall be discarded and a new specimen shall be run in its place.

8.1.3 Mount the wheels by placing the wheel marked "Left Hand This Side" on the left hand abrading head and the wheel marked" Right Hand This Side" on the right hand abrading head.

8.1.4 Before running the battery of tests, the ultrasonic cleaner shall be evaluated. This shall be done by by selecting the coated extra coated sample from Step 7.3, 7.5, and abrading.

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

8.1.4.2 Place the sample on the Taber Abraser, as described in 8.2.5

8.1.4.3 Set the Taber abraser for ten cumulative cycles and start the turntable.

8.1.4.4 Clean the sample for ten minutes with a fresh saline solution, using the ultrasonic cleaner to be used for the coating analysis.

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

8.1.4.6 Allow the sample to cool to room temperature before weighing

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

8.1.4.8 Repeat Steps 8.1.4.4-8.1.4.7 until the same mass (within balance error) is recorded for two consecutive cleanings.

8.1.4.9 Record the total number of cleanings used in 8.1.4.4-8.1.4.8.

8.1.4.10 Determine the required cleaning time necessary to obtain a stable mass, as (10*(*n*-1)) min, where *n* is the total number of cleanings determined in 8.1.4.9.

8.1.5 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.

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

8.1.7 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.

8.1.8 At the start of each new complete specimen run, the display indicating the number of cycles run shall be reset. This is done by pressing the "reset cycles completed" button. 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.

8.1.9 *Reference Standards*—Each group of six specimens shall include one unabraded "blank" specimen, as specified in 7.4. 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, and reweighed for as many cycles as would normally be used if abrasion testing were being performed. These values shall be logged as "blank" weight loss values.

8.2 *Operation*:

8.2.1 Clean the specimen for the time determined in 8.1.4.10 using an ultrasonic cleaner with saline solution. This solution shall be changed to a fresh solution before the first cleaning done that day.

8.2.2 Place the cleaned specimen in a 100°C oven for 10 min to dry.

8.2.3 Allow the specimen to cool to room temperature before weighing.

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

8.2.5 Place the specimen on the Taber™ abraser, coating side up and the turntable pin through the hole in the center of the specimen. Place the metal flange over the specimen, and secure the specimen and flange to the turntable with the screw fastener. Lower the abrading heads gently so that abrading wheels rest on the surface of the specimen. Lower the vacuum pick up after the abrading heads are resting on the coating surface.

8.2.6 Set the Taber™ abraser to reach the appropriate number of cumulative cycles (2, 5, 10, or 100). With the

vacuum set at 100 % power, start the Taber™ abraser in "vacuum only" mode. Let it run for no less than 5 s to allow vacuum to reach full power, then start the turntable rotation. When the turntable rotation for that partial run is completed, turn the vacuum off.

8.2.7 Clean, dry, cool, and weigh the specimen as before (see 8.2.1-8.2.4).

8.2.8 Repeat Steps 8.2.5-8.2.7 for 5, 10, and 100 cumulative cycles.

8.2.9 Repeat the steps specified in 8.2.1-8.2.8 for all specimens not reserved as blanks for the test.

8.2.10 Run each blank through the steps specified in 8.2.1-8.2.8 skipping the steps specified in 8.2.5 and 8.2.6.

9. Calculation

9.1 Calculate the cumulative mass 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 mass loss for n cycles,
$\langle w_o \rangle$ = average of three mass measurements at start the start of the test, and
$\langle w_n \rangle$ = average of three mass 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 8.1.4.10.

10.1.6 All cumulative mass losses associated with each specimen or blank following abrasion.

10.1.7 The mean cumulative mass loss and its standard of 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, appearance of discoloration of specimen, 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 E 691 (see Table 1). These tests were done for 2, 5, 10, and 100 cycles for a total of 12 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 E 691 min
Laboratories	6	6
Materials	3	3
Determinations	5	2

TABLE 2 Precision and Bias Data for the Full Study

Coating	Cycles	Average Weight Loss (g)	S_r (g)	S_R (g)	r (g)	R (g)
a	2	0.008 52	0.004 09	0.004 37	0.011 45	0.0122
b	2	0.011 66	0.003 92	0.006 24	0.010 97	0.0175
c	2	0.049 55	0.015 16	0.016 86	0.042 45	0.0472
a	5	0.014 83	0.005 44	0.006 08	0.015 23	0.017
b	5	0.020 77	0.006 34	0.007 53	0.017 76	0.0211
c	5	0.097 64	0.025 18	0.034 27	0.0705	0.0959
a	10	0.021 12	0.006 43	0.007 08	0.018	0.0198
b	10	0.030 2	0.007 92	0.008 86	0.022 17	0.0248
c	10	0.136 71	0.034 02	0.045 38	0.095 27	0.1271
a	100	0.054 06	0.012 22	0.017 17	0.034 22	0.0481
b	100	0.077 22	0.020 34	0.025 53	0.056 94	0.0715
c	100	0.274 51	0.063 06	0.066 52	0.176 57	0.1862

APPENDIX

(Nonmandatory Information)

X1. Rationale

X1.1 Abrasion may occur in implantable devices with metallic coatings produced by thermal spray processes. Excessive weight loss, however, is undesirable, as it may lead to third body wear of articulating surfaces.

X1.2 Although the use of sprayed flat specimens and the Taber™ abraser do not mimic the effect of bone and daily usage, this test method has been shown to be a reliable measure of the average wear/abrasion resistance of a thermal spray coating.

X1.3 Measurements are taken at 2, 5, 10, and 100 cycles. A minimum of two cycles was chosen to ensure that the turntable of the Taber™ abraser makes at least one complete rotation before any abrasion weight loss values are determined. After 100 cycles, which is the set maximum number of cycles, the mechanism of wear is no longer related to particle shedding.

X1.4 The ultrasonic cleaning solution used, as of yet, has had no corrosive effect on the coupons tested. Deionized water is used because it is both readily available and has a minimal amount of contaminants. A small amount of sodium chloride is added to the deionized water to neutralize the corrosiveness of the water. The amount of salt is well below the amount used for a physiologic saline solution and does not require that the specimens be rinsed with water after ultrasonic cleaning.