Designation: B 607 – 91 (Reapproved 2003)

Standard Specification for Autocatalytic Nickel Boron Coatings for Engineering Use[1]

This standard is issued under the fixed designation B 607; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 Nickel boron coatings are produced by autocatalytic (electroless) deposition from aqueous solutions. These solutions contain either an alkylamineborane or sodium borohydride as a reducing agent, a source of nickel ions, a buffer, complexant, and control chemicals.

1.2 This standard describes the requirements for coatings of autocatalytic nickel boron deposited from aqueous solutions onto substrates for engineering use. The specification classifies these coatings into two types:

1.2.1 *Type 1 coatings* have a boron content of 0.1 to less than 3.5 mass percent with the balance nickel.

1.2.2 *Type 2 coatings* have a boron content of 3.5 to 6 mass percent and a minimum of 90 mass percent nickel.

1.3 The coatings are hard and uniform in thickness, even on irregular shaped parts, and used in a wide range of applications.

1.4 Process solutions formulated with an alkylamineborane usually produce coatings that contain 0.1 to 3.5 % boron. Thin coatings of this type provide bondability and solderability on electronic components such as lead frames, electrical contacts, and headers. To maintain solderability, these coatings are generally not heat treated.

1.5 Process solutions formulated with sodium borohydride are strongly alkaline and are frequently used to plate steel and titanium parts to impart surface hardness and wear resistance properties. Deposits produced from these processes can contain 3 to 5 % boron and thallium or other metals which are used to stabilize the plating solution and modify the coating properties.

1.6 The physical and mechanical properties of these deposits such as density, hardness, stress, and melting point will vary with the boron content. The variation of boron content also affects the quantity and structure of nickel boride precipitated during heat treatment. In the as-plated condition the deposit consists of a predominantly amorphous mixture of nickel and boron with a hardness of about 700 HKN. When the deposit is heated above 300°C the nickel crystallizes, forming nickel clusters of Ni (111) and boron precipitates as nickel boride, Ni_3B (211) and (311), increasing the hardness to greater than 1000 HK_{100} for Type 2 coatings.

1.7 The nickel boron coatings are microporous and offer limited corrosion protection. Their columnar structure, however, is beneficial in reducing wear because it provides a means of trapping lubricants within the surface of the coated part.

1.8 This document describes only autocatalytic nickel boron coatings that have been produced without use of external electric sources.

1.9 The following hazards caveat pertains only to the Test Methods section of this specification: *This standard does not purport to address the safety problems 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.*

NOTE 1—The following AMS standards are not requirements. They are referenced for information only: AMS 2399 and AMS 2433.

2. Referenced Documents

2.1 *ASTM Standards:*

B 374 Terminology Relating to Electroplating[2]

B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section[2]

B 567 Test Method for Measurement of Coating Thickness by Beta Backscatter Method[2]

B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry[2]

B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings[2]

B 578 Test Method for Microhardness of Electroplated Coatings[2]

B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings[2]

B 656 Guide for Autocatalytic (Electroless) Nickel-Phosphorus Deposition on Metals for Engineering Use[2]

B 667 Practice for Construction and Use of a Probe for Measuring Electrical Contact Resistance[3]

B 678 Test Method for Solderability of Metallic-Coated Products[2]

[1] This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.01 on Engineering Coatings.

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[2] *Annual Book of ASTM Standards*, Vol 02.05.

[3] *Annual Book of ASTM Standards*, Vol 03.04.

B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings[2]

B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings[2]

D 2670 Test Method for Measuring Wear Properties of Fluid Lubricants (Falex Pin and Vee Block Method)[4]

D 2714 Test Method for Calibration and Operation of the Falex Block-on-Ring Friction and Wear Testing Machine[4]

E 39 Test Methods for Chemical Analysis of Nickel[5]

F 519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments[6]

2.2 *Aerospace Materials Specifications:*

AMS 2399 Electroless Nickel-Boron Plating[7]

AMS 2433 Electroless Nickel-Thallium-Boron Plating[7]

2.3 *U.S. Government Standards:*

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes[8]

MIL-STD-13165 Shot Peening of Metal Parts[8]

3. Terminology

3.1 *Definitions*—Many terms used in this standard are defined in Terminology B 374.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *cold shut*—a void on the surface which has been closed by machining and then partially opened through cleaning.

3.2.2 *hot halide stress-corrosion cracking*—a type of mechanical failure produced by halogenated solvents that have been absorbed onto titanium and then in the presence of heat cause microcracking, and the loss of mechanical strength.

3.2.3 *lap cracks*—a surface imperfection caused by cold working of steels producing a void which can be duplicated in the deposit.

3.2.4 *significant surface*—those substrate surfaces which the coating must protect and that are essential to the appearance.

4. Classification

4.1 The classification by type of these coatings establishes the amount of boron in the alloy.

4.1.1 *Type 1*—Coatings shall contain 0.1 to less than 3.5 mass percent boron with the balance nickel.

4.1.2 *Type 2*—Coatings shall contain 3.5 to 6 mass percent boron and a minimum of 90 mass percent nickel.

4.2 The classification by class of these coatings establishes the post treatment to be performed on the part(s). The post treatment steps are designed to reduce the potential for hydrogen embrittlement, increase the adhesion of the coating to the substrate, improve the fatigue properties of the part(s), and increase the wear resistance and hardness of the coating:

4.2.1 *Class 1*—Parts are supplied as plated with no post heat treatment.

4.2.2 *Class 2*—Parts are heat treated after plating to increase hardness. The coating is heat treated at 365 to 385°C for 90 min (see 7.2.4).

4.2.3 *Class 3*—Parts are heat treated after plating at 180 to 200°C for 2 to 23 h to improve coating adhesion on steel and for hydrogen embrittlement relief of steels (see 7.2.4).

4.2.4 *Class 4*—Parts are heat treated after plating at 120 to 130°C for a minimum of 1 h to improve adhesion on heat-treatable (age-hardened) aluminum alloys and carburized steels (see 7.2.4).

4.2.5 *Class 5*—Parts are heat treated after plating at 365 to 375°C for a minimum of 4 h to improve adhesion on titanium and titanium alloys (see 7.2.4).

4.3 The classification by grade establishes the minimum thickness of the coating:

4.3.1 *Grade A*—Parts are plated to a minimum coating thickness of 0.5 µm.

4.3.2 *Grade B*—Parts are plated to a minimum coating thickness of 12 µm.

4.3.3 *Grade C*—Parts are plated to a minimum coating thickness of 25 µm.

4.3.4 *Grade D*—Parts are plated to a minimum coating thickness of 75 µm.

5. Ordering Information

5.1 The purchaser should be aware of several processing considerations or options available to the processor and when ordering should supply the information described in 5.1.1 through 5.1.15 in the purchase order and drawings.

5.1.1 Title, ASTM designation, and year of issue of this specification.

5.1.2 Composition and metallurgical condition of the basis metal, assemblies of dissimilar materials must be identified.

5.1.3 Classification of the coating: type, class, and grade for this specification (see Section 4).

5.1.4 Minimum thickness required on the significant surface, and any maximum dimensions or tolerance requirements, if any (see 7.2.2).

5.1.5 Method of adhesion testing from Test Method B 571 to be used in acceptance requirements (see 8.3).

5.1.6 Requirements for certification and test reports (see Section 11).

5.1.7 Requirements for heat treatment of the part(s) for stress relief prior to plating (see 7.2.4).

5.1.8 Optional sampling plan for lot inspection of the part(s) (see 9.1 and 13.1).

5.1.9 Increased sampling frequency, if any, for qualification tests (see 7.3).

5.1.10 Supplemental requirements for shot peening of the part(s) (see 12.1).

5.1.11 Supplemental requirements for wear testing (see 12.2 and 12.3).

5.1.12 Supplemental requirements for heat treatment in vacuum or inert or reducing atmosphere (see 7.2.1 & 12.4).

5.1.13 Supplemental contact resistance requirements (see 12.5).

5.1.14 Supplemental solderability requirements (see 12.6).

[4] *Annual Book of ASTM Standards*, Vol 05.01.

[5] Discontinued; see *1994 Annual Book of ASTM Standards*, Vol 03.05.

[6] *Annual Book of ASTM Standards*, Vol 15.03.

[7] Available from Society of Automotive Engineers, Inc. (SAE), 400 Commonwealth Drive, Warrendale, PA 15096.

[8] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

5.1.15 Supplemental U.S. Government requirements, if any (see Section 13).

6. Materials and Manufacture

6.1 *Pretreatment*—Parts can be processed in accordance with Practice B 656.

6.1.1 A suitable method should be used to remove surface oxides and foreign materials which can cause poor adhesion and increased porosity.

6.1.2 A suitable method should be used to condition and activate the surface so that an adherent coating will be produced.

6.2 *Basis Material and Workmanship*— Nickel boron coatings will replicate the surface finish of the basis material. Imperfections in the surface of the basis material including scratches, porosity, pits, inclusions, roll and die marks, lap crack, burrs, cold shuts, and surface roughness that could adversely affect the coating should be brought to the attention of the purchaser prior to processing (see 7.2.1).

6.3 *Stress Relief*—Surface-hardened parts can require stress relief before plating. The stress relief heat treatment can reduce the hardness of some alloys and should therefore be reviewed by all parties before processing (see 5.1.7 and 7.2.4). Shorter times and higher temperature can be used if the resulting loss of surface hardness is acceptable to the purchaser.

6.4 *Hydrogen Embrittlement Relief*— Hydrogen embrittlement of high strength steels can be initiated by several different processing operations. Exposure of the parts to hydrogen sources will generally induce the condition. Care must be exercised whenever high strength steel is processed to ensure minimal exposure and timely relief treatment.

6.5 *Stress-Corrosion Cracking*—Titanium and titanium alloys are subject to stress-corrosion cracking after processing. Pretreatment solutions including rinses should not contain methanol, halogenated hydrocarbon, or more than 50 ppm chlorides, all of which can cause subsequent stress-corrosion cracking when the parts are heated to 260°C or higher.

7. Requirements

7.1 *Process*—The nickel boron coatings shall be produced by autocatalytic nickel deposition from aqueous solutions.

7.2 *Acceptance Requirements*—The acceptance requirements in 7.2.1 through 7.2.4 are required for all lots of part(s). Each lot of part(s) shall be sampled with the recommended procedure described in Section 9 of this specification.

7.2.1 *Appearance*—The coating shall have a uniform appearance without visible imperfections such as blisters, pits, pimples, and cracks.

7.2.1.1 Imperfections that arise from the surface condition of the basis metal and that cannot be removed using conventional metal finishing techniques and that persist in the coating shall not be cause for rejection.

7.2.1.2 Discoloration caused by heat treatment shall not be cause for rejection unless specified in the ordering information (see 5.1.12 and 12.4).

7.2.2 *Thickness*—The coating thickness shall be measured and conform to the specified grade.

7.2.3 *Adhesion*—The coating shall pass the adhesion test of Test Method B 571 as specified in the ordering information (see 5.1.15).

7.2.4 *Heat Treatment*:

7.2.4.1 All steel part(s) with a tensile strength of 1000 MPa or greater shall be heat treated at 190 ± 15°C for stress relief in accordance with Table 1 before plating and baked within 3 h after plating for hydrogen embrittlement relief.

7.2.4.2 Class 2 coated part(s) shall be heat treated after plating in accordance with Table 2 for precipitation hardening of the deposit.

7.2.4.3 Heat treatment for Class 3 coated steel part(s) shall be in accordance with Table 1.

7.2.4.4 Heat treatment for Class 4 and 5 coated part(s) other than steel basis material shall be in accordance with Table 3.

7.3 *Qualification Requirements*—Coating and process attributes that require testing on a monthly basis, or more frequently when specified in the ordering information by the purchaser. A test specimen or part, processed in a manner that duplicates the characteristics of production parts, shall be produced and used in these tests.

7.3.1 *Hardness*—The hardness of the Type 2, Class 2, Grade C and D coating shall be not less than 1000 HK_{100} as measured by Test Method B 578.

7.3.2 *Composition*—The coating composition produced from the process shall be analyzed for nickel and boron. The alloy produced shall be within the range specified for the coating type.

7.3.3 *Hydrogen Embrittlement*—The process and coating shall be evaluated for freedom from hydrogen embrittlement and pass requirements of Test Method F 519.

8. Test Methods

8.1 *Test Specimens*:

8.1.1 When separate test specimens are required, the number to be used, the material from which they are to be made, and their shape and size shall be specified by the purchaser.

8.1.2 When separate test specimens are used for acceptance or qualification testing of the coating, the specimens shall be made of the same material as the part(s), have the same metallurgical condition as the part(s), and be processed with the part(s).

8.2 *Thickness*—The thickness shall be measured at any place on the significant surface designated by the purchaser, and the measurement shall be made with an accuracy of better than 10 % by a method selected by the purchaser. Fig. X1.1 describes the density of these coatings in relationship to their boron content.

TABLE 1 Heat Treatment for Stress Relief Before Plating and for Hydrogen Embrittlement Relief After Plating

NOTE 1—Heat treatment for stress relief of surface hardened steels prior to plating shall be 140 ± 10°C for 5 h.

Tensile strength steel, MPa	Heat treatment, minimum, at 190 ± 15°C, h
1000 to 1450	2
1450 to 1800	18
over 1800	23

TABLE 2 Heat Hardening of Nickel Boron Deposits

Temperature, °C	Time, min
375 ± 10	90

TABLE 3 Post Heat Treatment for Class 4 and 5 Coatings

Class	Temperature, °C	Time, minimum, h
Class 4	125 ± 5	1
Class 5	370 ± 5	4

8.2.1 *Weigh, Plate, Weigh Method*—Using a similar substrate material, weigh to the nearest milligram before and after plating. Calculate the thickness from the increase in mass, surface area, and density of the coating.

NOTE 2—The density of the coating will decrease as the mass percent boron in the coating increases. For Type 1 coatings the density is approximately 8.7 g/cm^3 and for Type 2 coatings it is approximately 8.2 g/cm^3 (see Appendix X1).

8.2.1.1 *Example*:

$$\text{Thickness, µm} = \frac{10 \times W}{A \times D} \tag{1}$$

where:
W = mass gain, mg,
A = area of plating, cm^2, and
D = density of deposit, g/cm^3.

8.2.2 *Metallographic Sectioning*—Plate a specimen of similar composition and metallurgical condition to the part(s) being plated, or use a sample from the lot, cross-section, mount, and polish. Using a calibrated Vernier microscope, examine the thickness of the deposit and average over 10 readings using Test Method B 487.

NOTE 3—Microscopic metallographic sectioning is dependent on the sample preparation.

8.2.3 *Micrometer Method*—Measure the part(s) or test a coupon in a specific area before and after plating using a suitable micrometer. Ensure that the specimen is at the same temperature for each measurement and that the surface measured is smooth.

8.2.4 *Beta Backscatter Method*—The coating thickness can be measured by the use of a beta backscatter device using Test Method B 567. This technique measures the mass per unit area of the coating applied over the substrate and is displayed as thickness. The use of the beta backscatter method is restricted to basis metals that have an atomic number less than 18 or greater than 40. The instrument shall be calibrated with specimens having the same basis material and coating composition as the part(s).

8.2.5 *X-Ray Spectrometry*—The coating thickness can be measured by X-ray spectrometry using Test Method B 568. This technique measures the mass per unit area of the coating applied over the substrate and is displayed as thickness. It can only be used for coatings of known boron content. The instrument shall be calibrated with specimens having the same basis material and coating composition as the part(s).

8.3 *Adhesion*—Adhesion of the coating to the basis material shall be evaluated using one of the procedures described in Test Method B 571 (see 5.1.5).

8.4 *Composition*:

8.4.1 *Inductively Coupled Plasma*—Chemical analysis by inductively coupled plasma—Dissolve approximately 50 mg of a foil sample in 50 mL of 100 % warm reagent grade nitric acid. Analyze the solution for nickel at 232.00 nm, boron at 241.77 nm, and any other alloying element at an appropriate wavelength. From the results of the analysis calculate a fraction of the boron or alloying element divided by the total of boron, nickel, and alloying element. Report the percent boron and alloying element if present in the coating.

8.4.2 *Other Chemical Methods*—Determine mass percent nickel content according to Methods E 39 on known weight of deposit dissolved in nitric acid. The determination of mass percent boron content may be accomplished by an appropriate analytical method (such as atomic absorption spectroscopy). Report the percent boron and alloying elements if present in the coating.

9. Sampling

9.1 The sampling plan used for the inspection of a quantity of coated parts (lot) shall be as described in Test Method B 602, unless otherwise specified by purchaser in the purchase order or contract (see 5.1.8 and 13.1).

NOTE 4—Usually, when a collection of coated parts (the inspection lot; see 9.2) is examined for compliance with the requirements placed on the parts, a relatively small number of parts—the sample—is selected at random and inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one for use with tests that are destructive. The purchaser and producer may agree on the plan(s) to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance on the selection of a plan. When Guide B 697 is specified, the purchaser and producer need to agree on the plan to be used.

Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The purchaser and producer may agree on the plan(s) to be used. If they do not, Method B 762 identifies the plan to be used.

9.2 An inspection lot shall be defined as a collection of coated parts that are of the same kind, that have been produced to the same specification, that have been coated by a single producer at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

9.3 If separate test specimens are used to represent the part(s) in the test, the number shall be that required in 8.1.

10. Rejection and Rehearing

10.1 Part(s) that fail to conform to the requirements of this standard may be rejected. Rejection shall be reported to the producer promptly in writing. In the case of dissatisfaction with the results of a test, the producer may make a claim for a rehearing. Coatings that show imperfections may be rejected.

11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that the samples representing each lot have been processed, tested, and inspected as directed in this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Supplementary Requirements

12.1 *Shot Peening*—When specified by the purchaser in the ordering information, the part(s) shall be shot peened prior to plating in accordance with MIL-STD-13165.

12.2 *Abrasive Wear*—When specified by the purchaser in the ordering information, the coating shall be tested for abrasion wear resistance using the procedure described in Appendix X2 of this specification. The coating shall meet a maximum wear rate which is specified by the purchaser and agreed to by the producer.

12.3 *Adhesive Wear*—When specified by the purchaser in the ordering information, the coating shall be tested for adhesive wear resistance using Test Method D 2714 or D 2670. The wear rate shall be specified by the purchaser and agreed to by the producer.

12.4 *Inert Atmosphere*—When specified by the purchaser in the ordering information, the coating shall be heat treated in a vacuum or an inert or reducing atmosphere to prevent surface oxidation of the coating.

12.5 *Contact Resistance*—When specified by the purchaser in the ordering information, the coating shall be tested for contact resistance using Practice B 667.

12.6 *Solderability*—When specified by the purchaser in the ordering information, the unaged coating shall pass Test Method B 678.

13. Special Government Regulations

13.1 *Sampling*—The producer when plating part(s) for the U.S. government and military use shall use a sampling plan from MIL-STD-105.

13.2 *Shot Peening*—High strength steel part(s) processed for U.S. government and military use shall be shot peened in accordance with MIL-STD-13165.

APPENDIXES

(Nonmandatory Information)

X1. DENSITY OF NICKEL BORON COATINGS

X1.1 Many thickness test methods measure the mass per unit area and display the thickness in linear units. These test methods require a density factor to calculate the thickness. Fig. X1.1 can be used to determine the density of nickel coatings with varying boron contents.

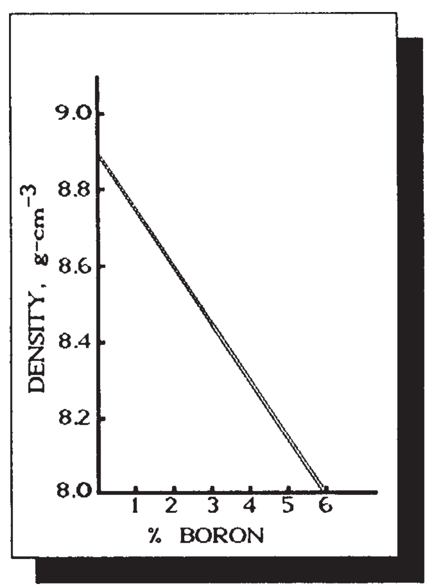

FIG. X1.1 Density of Nickel Coatings with Varying Boron Content

X2. TABER ABRASER WEAR TEST METHOD

X2.1 Scope

X2.1.1 This test method will evaluate the resistance of the coating to abrasive wear. The test is performed on a rotating plated panel that is abraded by two rotating rubber wheels. The panel is weighed before and after each 1000 cycles for weight loss determination. Duration of the test is 5000 cycles and it can be extended to 25 000 cycles.

NOTE X2.1—Variations in results have been attributed to the humidity in the laboratory and the storage conditions of the CS-10 wheels. Care should be taken to control the humidity between tests as recommended by the equipment manufacturer.

X2.1.2 The results are variable between tests and therefore three plated test specimens should be tested to 6000 cycles each. The results should be averaged without the first 1000 cycles and the abrasion wear resistance reported.

X2.2 Apparatus [9]

X2.2.1 *Taber Abraser Wear Testing Unit*—The unit must be capable of loading with a 1000 g load and operating with a vacuum.

X2.2.2 *Abrasive Wheels*— Use CS-10 (resilient rubber) Taber wheels. To reface the wheels use CS-11 discs.

X2.2.3 *Test Specimens*— Test specimens shall be made from 20 gage CR steel 4 by 4 in. (100 mm by 100 mm by 1.3 mm) with a 0.250-in. (6.35-mm) hole in the center.

X2.2.4 *Analytical Balance*, Capable of weighing 150 g ± 0.1 mg.

[9] The Taber Abraser Wear Testing Unit, abrasive wheels and discs, and test specimens are available from Teledyne Taber, North Tonawanda, NY 14120.

X2.3 Procedure

X2.3.1 Plate three specimens with 0.001 in. (25 μm) of nickel boron coating.

X2.3.2 Heat treat the specimens for 375 ± 10°C for 90 min.

X2.3.3 Abrasion test the specimens. For each of the three specimens follow X2.3.3.1 through X2.3.3.6:

X2.3.3.1 Smooth the coating for 1000 cycles to remove any surface roughness. The wheels shall be loaded with 1000 g with the vacuum on high for the entire test.

X2.3.3.2 Cool and weigh the specimen to the nearest 0.1 mg.

X2.3.3.3 Dress the CS-10 wheels with a CS-11 disc for 50 cycles.

X2.3.3.4 Abrasion test the coating with a 1000 g load for 1000 cycles.

X2.3.3.5 Repeat X2.3.3.2, X2.3.3.3, and X2.3.3.4 until a total of 6000 cycles have been accomplished for each specimen.

X2.3.3.6 Determine the relative humidity in the test area and report it with the wear results.

NOTE X2.2—This equipment should be operated using manufacturer's recommended procedures.

X2.4 Reporting

X2.4.1 Determine the average weight loss in milligrams for each specimen per 1000 cycles (Taber Wear Index) and the mean weight loss per 1000 cycles for all specimens. Report the mean and standard deviation for the coating and the humidity during testing.