Designation: B 839 – 94 (Reapproved 2003)

Standard Test Method for Residual Embrittlement in Metallic Coated, Externally Threaded Articles, Fasteners, and Rod—Inclined Wedge Method[1]

This standard is issued under the fixed designation B 839; 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.

INTRODUCTION

When atomic hydrogen enters steels and certain other alloys, it can cause loss of ductility or load carrying ability or cracking (usually as submicroscopic cracks), or catastrophic brittle failures at applied stresses well below the yield strength or even the normal design strength for the alloys. This phenomenon often occurs in alloys that show no significant loss in ductility, when measured by conventional tensile tests, and is frequently referred to as hydrogen-induced delayed brittle failure, hydrogen stress cracking, or hydrogen embrittlement. The hydrogen can be introduced during cleaning, pickling, phosphating, electroplating, autocatalytic processes, and in the service environment as a result of cathodic protection reactions or corrosion reactions. Hydrogen can also be introduced during fabrication, for example, during roll forming, machining, and drilling due to lubricant breakdown as well as during welding or brazing operations.

1. Scope

1.1 This test method covers the determination of, on a statistical basis, the probability of the existence of hydrogen embrittlement or degradation in:

1.1.1 A batch of barrel electroplated, autocatalytic plated, phosphated, or chemically processed threaded articles or fasteners and

1.1.2 A batch of rack plated threaded articles, fasteners, or rod.

1.2 Industrial practice for threaded articles, fasteners, and rod has evolved three graduated levels of test exposure to ensure reduced risk of hydrogen embrittlement (see Section 3). These levels have evolved from commercial applications having varying levels of criticality. In essence, they represent the confidence level that is required. They also represent the time that finished goods are held before they can be shipped and used. This time equates to additional cost to the manufacturer that may of necessity be added to the cost of the finished goods.

1.3 This test method is applicable to threaded articles, fasteners, and rod made from steel with ≥1000 MPa (with corresponding hardness values of 300 $HV_{10\ kgf}$, 303 HB, or 31 HR_c) or surface hardened threaded articles, fasteners, or rod.

1.4 This test method shall be carried out after hydrogen embrittlement relief heat treatment in accordance with the requirements of Guide B 850. It may also be used for assessing differences in processing solutions, conditions, and techniques. This test method has two main functions: first, when used with a statistical sampling plan it can be used for lot acceptance or rejection, and second, it can be used as a control test to determine the effectiveness of the various processing steps including pre- and post-baking treatments to reduce the mobile hydrogen in the articles, fasteners, or rod. While this test method is capable of indicating those items that are embrittled to the extent defined in Section 3, it does not guarantee complete freedom from embrittlement.

1.5 This test method does not relieve the processor from imposing and monitoring suitable process control.

1.6 This test method has been coordinated with ISO/DIS 10587 and is technically equivalent. (**Warning—**Great care should be taken when applying this test method. The heads of embrittled articles, fasteners, or rod may suddenly break off and become flying projectiles capable of causing blindness or other serious injury. This hazard can occur as long as 200 h after the test has started. Hence, shields or other apparatus should be provided to avoid such injury.)

[1] This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.

Current edition approved May 10, 2003. Published July 2003. Originally approved in 1994. Last previous edition approved in 1994 as B 839 – 94.

NOTE 1—The use of inhibitors in acid pickling baths does not necessarily guarantee avoidance of hydrogen embrittlement.

1.7 *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:*

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

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

F 436 Specification for Hardened Steel Washers[3]

B 850 Guide for Post-Coating Treatments of Steel for Reducing the Risk of Hydrogen Embrittlement[2]

2.2 *ISO Standards:*

ISO/DIS 10587 Residual Embrittlement in Metallic Coated, Externally Threaded Articles, Fasteners and Rod—Inclined Wedge Method[4]

ISO 4519 Electrodeposited Metallic Coatings and Related Finishes—Sampling Procedures for Inspection by Attributes[4]

2.3 *Military Standard:*

MIL-STD-1312 Fastener Test Methods[5]

3. Terminology

3.1 *Definitions*—For the purposes of this test method the following definitions apply:

3.1.1 *batch*—a distinct portion of items processed collectively as a single group through the same identical treatment steps at the same time on the same rack or in the same barrel.

3.1.2 *embrittled*—where parts fail immediately or up to 48 h in test.

3.1.2.1 *Discussion*—The degree to which parts within a single plated batch or a given lot can be embrittled can vary over a wide range. The degree of embrittlement is a function of the concentration of atomic hydrogen in the individual parts in the batch or lot, measured in parts per million, and in particular that portion of the hydrogen that is mobile or free to migrate to areas of high stress concentration.

3.1.3 *Grade 48 proof*—where there are no failures after 48 h of test.

3.1.4 *Grade 96 proof*—where there are no failures after 96 h of test.

3.1.5 *Grade 200 proof*—where there are no failures after 200 h of test.

3.1.6 *lot*—a group of items processed through the same or similar steps at the same time or over a contiguous time period and from the same heat of material. The lot may be broken down into a number of batches for processing purposes and then reassembled into the same lot.

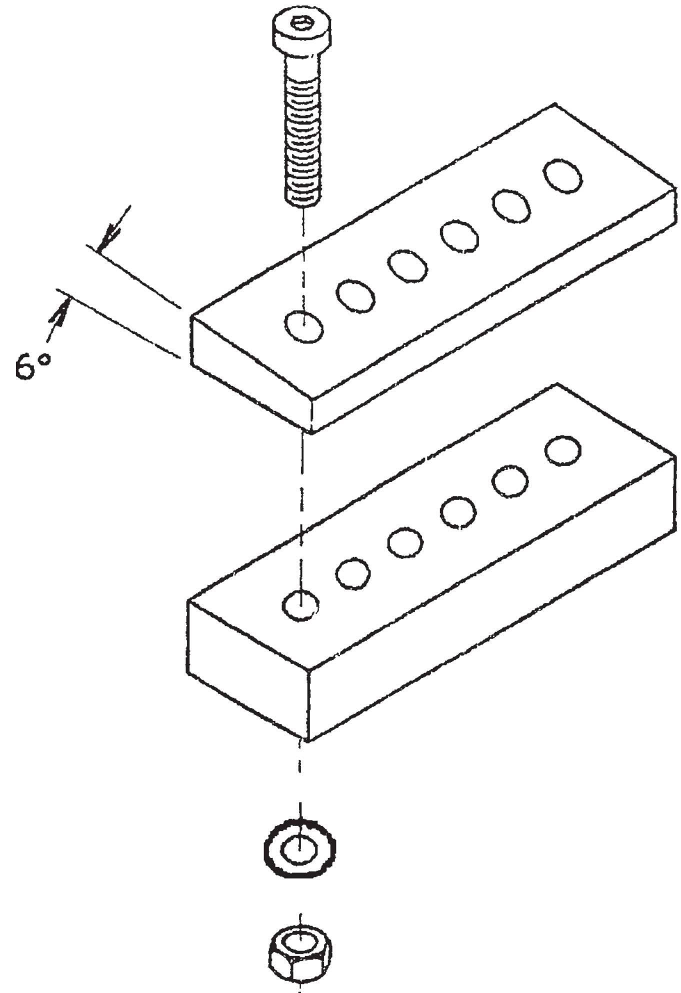

FIG. 1 Example of 6° Wedge and Parallel Filler Plate

4. Summary of Test Method

4.1 The threaded articles, fasteners, or rod are subjected to stress by tensioning with a mating nut after insertion through a clearance hole in a hardened rectangular wedge of steel; see Fig. 1. Additional hardened rectangular pieces of steel with parallel faces are provided as filler plates and are inserted so that the required length of the threaded article is placed under test. Other loading systems and fixtures are permissible as long as the same load, angle, and exposure are created for the test. The upper surface of the wedge is ground at an angle to the lower surface. The mating nut is tensioned by any means capable of measuring tensile load. The torque method described in 6.4 is one such method. If the torque method of tightening is used, the fasteners are torqued to the desired value, held for the minimum specified hours, and then checked to determine if the initial torque has been maintained. Following this they are examined for embrittlement failures. See Section 9.

NOTE 2—Increasing the applied torque by a small percentage as a safety factor is not recommended.

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

[3] *Annual Book of ASTM Standards*, Vol 01.08.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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

TABLE 1 Wedge Angle Selection (in degrees)

Nominal Size of Threaded Article	Articles with Unthreaded Lengths Less than 2 Diameters	Articles with Unthreaded Lengths 2 Diameters and Longer
2 to 6 mm	6	6
1⁄16 to 1⁄4 in.	6	6
6 to 18 mm	4	6
1⁄4 to 3⁄4 in.	4	6
over 18 to 38 mm	0	4
over 3⁄4 to 1½ in.	0	4

5. Significance and Use

5.1 The use of this test method can significantly reduce the risk of sudden catastrophic failure of threaded articles and fasteners, below their design strength, due to hydrogen embrittlement.

6. Apparatus

6.1 *Test Fixture* comprising a hardened wedge (see Fig. 1), one or more filler plates, and a hardened washer. The hole in each shall be as close to the major diameter of the threaded article, fastener, or rod being tested as practical. Excess clearance space may cause the fastener to tilt in the hole and can result in a failure at a lower torque value.

6.2 *Fixture With Multiple Holes* has been found useful for multiple or repetitive testing. The fixture can be readily made from a rectangular piece of an air hardening grade of steel with one face ground to the appropriate wedge angle and hardened to HR_c 60.

6.3 *Wedge*—Shall have an angle as specified in Table 1.

6.4 *Filler Plate(s)*—Shall be of the same steel grade and hardness as the wedge fixture and have a thickness such that, after installation and tightening, a minimum of three full threads of the test fastener will be engaged and no more than five full threads will extend beyond the nut.

6.5 *Washer*—Shall be HR_c 38 to 45 and shall conform to the requirements of Specification F 436.

6.6 *Torque Application Device*—If the torque method of tightening is used, the tightening torque shall be determined using a load measuring device capable of measuring the actual tension induced in the article, fastener, or rod as the item is tightened.

6.7 *Torque Determination*—Five items from the test lot shall be selected at random. Each shall be assembled into the load measuring device, mated with a nut, and the nut tightened until a load equal to 75 % of the ultimate tensile strength of the item is induced. The torque required to induce this load shall be measured and the arithmetic average of the five measured torques shall be the tightening torque. Calculated torque versus tension methods of testing such as the $T = KDL$ formula used in MIL-STD-1313 are not sufficiently accurate for use in this test and shall not be used.

7. Sampling

7.1 The document specifying this test method shall specify an AQI level and sampling plan to be used. Guidance in the selection of sampling plans is provided in Guide B 697. Widely used sampling plans are provided in Test Method B 602 and its equivalent ISO 4519.

7.2 A minimum sample size of 30 pieces is necessary from each embrittlement relief treated batch that exceeds 500 pieces plated as a single group.

8. Procedure

8.1 *Test Item Placement*—Place the test items in the clearance holes with the heads positioned against the angle of the wedge. In the case of items with square, hexagonal, or similar straight side heads, a straight side shall be placed against the angle of the wedge. In the case of elliptical or other shaped heads, the side with the minor radius of the ellipse shall be placed against the angle of the wedge. In the case of items without heads, studs, or threaded rod, one end shall be nutted and tested as the head. When the items are threaded with different pitch threads, the finer thread shall be treated as the head. Nut the free end of the items and run them up finger-tight. No significance has been found between the start of the thread on an article in relation to the angle of the wedge.

8.2 *Torque Application*—Clamp the wedge device with the nutted ends facing in a convenient position in a securely attached vice. Using a calibrated torque tool tighten the nuts to the desired torque and record the values. The wedge should be removed from the vice and left undisturbed for the test period. See Section 3.

9. Evaluation

9.1 *Cracks, Separated Heads, and Breakage*—After the specified holding period is complete, examine each item for failures such as cracks, separated heads, and breakage. Use finger pressure to check each head for breakage. Cracks can be identified by examination at 10× magnification, magnetic particle inspection, or the use of a liquid dye penetrant.

9.2 *Relaxed Torque*—Following the examination of the specimens in 9.1, place the wedge in a vice and carefully turn each mating nut, with the torque tool, in the *on* direction until a forward angular motion, after break loose, is noticeable. Record the torque value at break loose and compare it with the initially recorded torque. Torque relaxation greater than 10 % shall be recorded as failure. Remove the nuts and examine the items for transverse cracks, which shall also be recorded as failure.

10. Report

10.1 Report the following information:

10.1.1 ASTM designation number of this test method,

10.1.2 Batch identification number and total number of parts in the batch,

10.1.3 Number of parts tested,

10.1.4 Number of broken parts, parts with visible cracks or other observed failures, and parts that exhibited relaxed torque, and

10.1.5 Duration of the test method.

11. Precision and Bias

11.1 *Precision*—The precision of this test method has not been determined.

11.2 *Bias*—The bias has not been determined.

12. Keywords

12.1 hydrogen embrittlement test; metallic coated; residual embrittlement test; testing threaded articles; threaded fasteners; threaded rod

APPENDIX

(Nonmandatory Information)

X1. SOURCES OF INTRODUCTION OF HYDROGEN INTO THREADED ARTICLES

X1.1 The preparation and metallic coating of threaded articles, fasteners, and rod are usually accomplished by the barrel-plating process. In this process, quantities of an item are placed within a containment vessel, called a barrel. The barrel is designed to move the group of items, together, through each of the process steps, allowing ready ingress and egress of processing solutions and rinses. As the barrel is moved through the process steps, it is also rotated such that the individual items are constantly cascading over one another. In some of the process steps, notably the electrocleaning and electroplating steps, an electric current is applied to the group of items. The cascading action randomly exposes the surfaces of each individual piece to the process electrodes while also maintaining electrical continuity among all the parts.

X1.2 During both the electrolytic and non-electrolytic steps hydrogen is generated and exposed to the individual items in the same random manner. Experience and experimentation have shown that despite the best practice, some individual items of the group will receive more hydrogen exposure than others of the group due to the randomness of the barrel-plating process.

X1.3 Examination and analysis of barrel-plated items have shown that when hydrogen charging of such items does occur, it follows the normal distribution or bell-shaped curve. A very few of the items absorb no hydrogen, the vast majority absorb a small amount of hydrogen, and a very few items absorb more hydrogen. Baking treatment, which can vary in time and temperature, can render the normally mobile hydrogen immobile, thus rendering the individual items free of hydrogen embrittlement. However, a number of variables exist within processes that, despite the best practice, increase hydrogen charging on the parts. Platers cannot eliminate or easily control such random hydrogen charging. Therefore, testing representative quantities of the finished items, selected using a statistical sampling plan, is necessary. Thus, it is not always possible to guarantee that lots of threaded articles produced by such processes are completely free of hydrogen embrittlement. Rather they can only guarantee that representative quantities of the lot have been tested and have shown no hydrogen embrittlement failures for the specified period of test.