



Designation: F 1113 – 87 (Reapproved 1999)

Standard Test Method for Electrochemical Measurement of Diffusible Hydrogen in Steels (Barnacle Electrode)¹

This standard is issued under the fixed designation F 1113; 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 describes the procedure for measuring diffusible hydrogen in steels by an electrochemical method.

1.2 This test method is limited to carbon or alloy steels, excluding austenitic stainless steels.

1.3 This test method is limited to flat specimens to which the cell can be attached (see 4.6 and 4.8).

1.4 This test method describes testing on bare or plated steel after the plate has been removed (see 4.4).

1.5 This test method is limited to measurements at room temperature, 20 to 25°C (68 to 77°F).

1.6 *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:

A 165 Specification for Electrodeposited Coatings of Cadmium on Steel²

B 183 Practice for Preparation of Low-Carbon Steel for Electroplating³

B 242 Practice for Preparation of High-Carbon Steel for Electroplating³

D 1193 Specification for Reagent Water⁴

F 519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments⁵

G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing⁶

¹ This test method is under the jurisdiction of ASTM Committee F-7 on Aerospace and Aircraft and is the direct responsibility of Subcommittee F07.04 on Hydrogen Embrittlement.

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² Discontinued in 1988 and replaced by Specification B 766.

³ Annual Book of ASTM Standards, Vol 02.06.

⁴ Annual Book of ASTM Standards, Vol 11.01.

⁵ Annual Book of ASTM Standards, Vol 15.03.

⁶ Annual Book of ASTM Standards, Vol 03.02.

3. Summary of Test Method

3.1 A hydrogen-containing part is made the anode in an electrochemical cell. The diffusible (atomic) hydrogen, which comes to the metal-electrolyte interface, is oxidized to protons (H⁺); H⁺ combines with hydroxyl ions (OH⁻) in the electrolyte to form water. The oxidation current is measured and related to the hydrogen concentration.

4. Significance and Use

4.1 The critical level of hydrogen in steels is that hydrogen which can build up to high concentrations at points of high triaxial stress causing embrittlement of the steel which can lead to catastrophic damage. This hydrogen can enter by various means, such as during pickling and electroplating. Means of reducing this hydrogen during processing are given in Specification A 165 and Practices B 183 and B 242. It is still necessary, however, to know how effective these methods are. Though the ultimate reason for measuring this hydrogen is to relate it to embrittlement, this is not within the scope of this test method. As susceptibility to hydrogen embrittlement is a function of alloy type, heat treatment, intended use, and so forth, the tolerance for hydrogen must be determined by the user according to Method F 519.

4.2 Though the actual hydrogen concentration is not determined in this test method, the current densities have been shown to be useful as an indication of relative hydrogen concentrations (1-3),⁷ and therefore the degree of hydrogen embrittlement (1,2). Thus, measurements can be compared to one another (see 4.1 and 7.1).

4.3 This test method is applicable as a quality control tool for processing (such as to monitor plating and baking) or to measure hydrogen uptake caused by corrosion.

4.4 This test method is nondestructive; however, if there is a coating, it must be removed by a method which has been demonstrated to neither damage the steel nor introduce hydrogen to make the measurement.

⁷ Boldface numbers in parentheses refer to the list of references at the end of this standard.

4.5 This test method is also applicable to situations producing continuous hydrogen permeation, such as high pressure hydrogen cylinders or corrosion processes. The results, however, would require a different treatment and interpretation (4).

4.6 This test method is also applicable to small parts, such as fasteners. The technique, procedure, and interpretation would, however, have to be altered.

4.7 Use of this test method on austenitic stainless steels and other face centered cubic (FCC) alloys would require different measurement times and interpretation of results because of differing kinetics.

4.8 This test method can be used on slightly curved surfaces as long as the gasket defines a reproducible area. The area calculation must, however, be changed.

5. Apparatus

5.1 *Cell*—A photo and drawing of a typical cell, which has been found to be acceptable for hydrogen measurements, are shown in Figs. 1 and 2, respectively.

5.1.1 The cell is made of a nonmetallic material that will not react with or contaminate the solution. The side opening has a recess to accommodate the silicone rubber gasket.

5.1.2 *Gasket*, silicone rubber, shall provide a reproducible solution-contact area with the specimen, preferably 1.0 cm^2 .

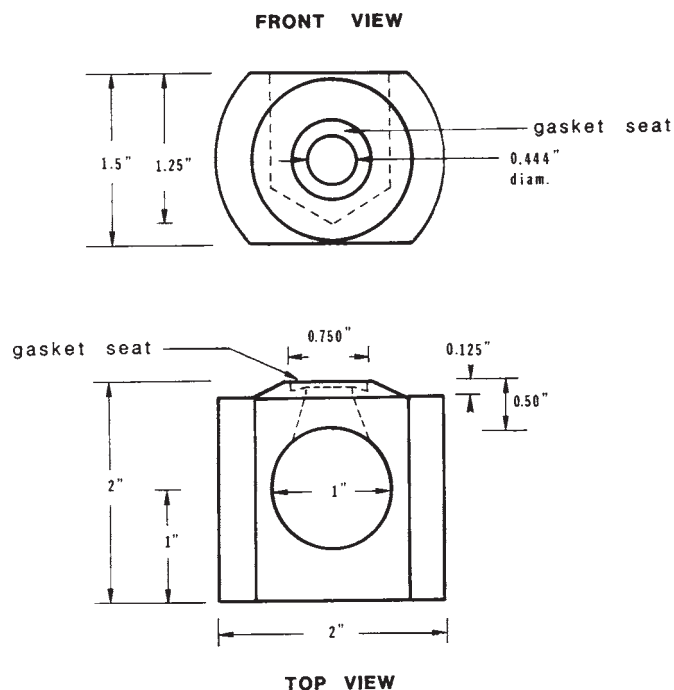


FIG. 2 Engineering Drawing of Cell (3)

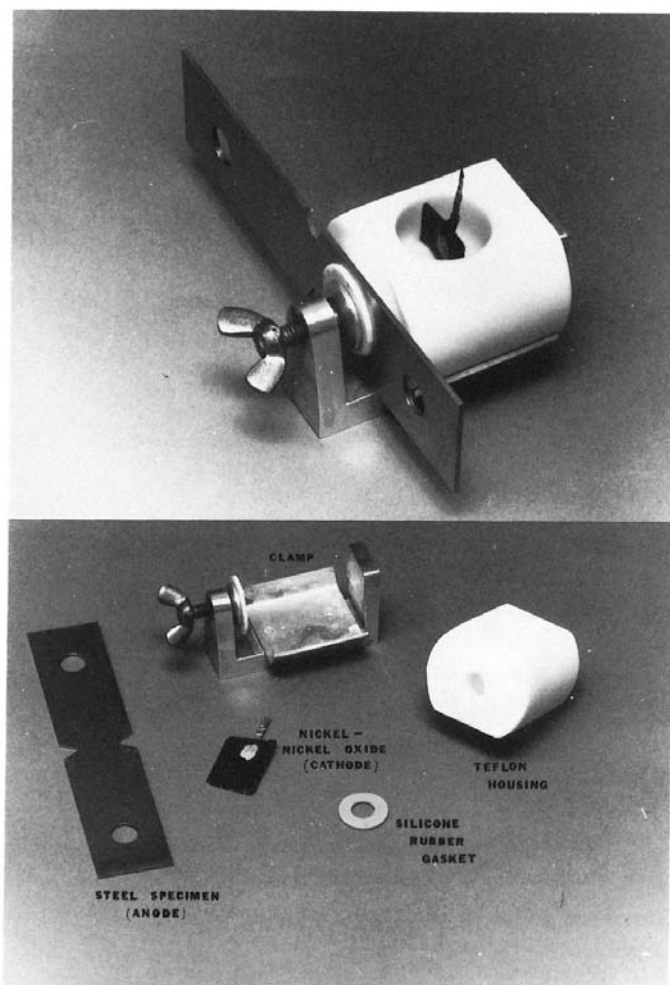


FIG. 1 Photograph of Cell

5.1.3 *Cell Holder*, a cradle-like C-clamp. Other clamping devices can be used if necessary, such as for larger parts.

5.1.4 *Cathode*, a nickel/nickel oxide electrode. It is made by removing the positive plate from a nickel/cadmium battery and attaching a nickel wire or foil. The area of this cathode shall be approximately five times that of the anode.

5.1.5 *Anode*—The anode is the specimen.

5.1.6 The cell is left open to the atmosphere. No purging is used.

5.2 *Current Measuring Device*—The current can be measured by any method that will not affect its value. A zero resistance ammeter (5), a current follower (6), and the current measuring system shown in Fig. 3 (1) have been found to be acceptable. The following description refers to Fig. 3.

5.2.1 *Standard Resistor*, connected across the cell through a switch.

5.2.2 *Electrometer*, to determine the current by measuring the voltage drop across the resistor. A 10-k Ω resistor with an

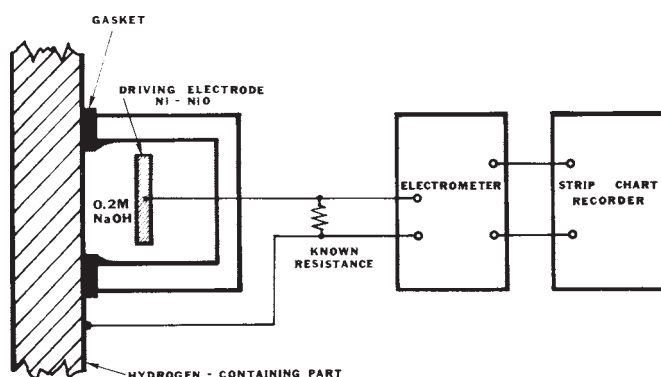


FIG. 3 Schematic of Measuring Apparatus (1)