Designation: F326 – 23

Standard Test Method for Electronic Measurement for Hydrogen Embrittlement From Cadmium-Electroplating Processes[1]

This standard is issued under the fixed designation F326; 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 covers an electronic hydrogen detection instrument procedure for measurement of plating permeability to hydrogen. This method measures a variable related to hydrogen absorbed by steel during plating and to the hydrogen permeability of the plate during post plate baking. A specific application of this method is controlling cadmium-plating processes in which the plate porosity relative to hydrogen is critical, such as cadmium on high-strength steel.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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.* For specific hazard statement, see Section 8.

1.4 *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:*[2]

D1193 Specification for Reagent Water

F519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *hydrogen pressure peak*—the maximum hydrogen pressure value (see I_H) obtained when the probe is heated following calibration, plating, or fluid testing.

3.2 *Symbols:*

3.2.1 *HP* = calibration hydrogen pressure peak.

3.2.2 HP_p = plating hydrogen pressure peak.

3.2.3 I_E or I_e = probe cathode emission current.

3.2.4 I_H = probe hydrogen pressure.

3.2.5 I_y = integral of I_H curve from probe on to *HP*.

3.2.6 *lambda* = time in seconds for hydrogen pressure peak to drop to half its value.

3.2.7 λ = lambda obtained from a calibration run.

3.2.8 λ_p = lambda obtained from a plating run.

3.2.9 λ_{pc} = normalized test lambda, obtained as follows:

$$\lambda_{pc} = \lambda_p\,(40/\lambda) \tag{1}$$

3.2.10 $\bar{\lambda}_{pc}$ = arithmetic average of normalized lambdas for a set of tests.

3.2.11 *range* = difference between maximum λ_{pc} and minimum λ_{pc} for a given set of tests.

3.2.12 *run* = calibration or plating of a probe.

3.2.13 *test* = single evaluation of a plating solution for hydrogen embrittlement determination; run using a previously calibrated probe.

3.2.14 *set of tests*—all consecutive tests on a plating solution for a given operator-instrument-day evaluation.

3.2.15 *window*—test surface of a probe described in Fig. 1(A).

4. Summary of Test Method

4.1 This method uses a metal-shelled vacuum probe as an ion gage to evaluate electrodeposited cadmium characteristics relative to hydrogen permeation. After calibration, a section of the probe shell is electroplated at the lowest current density encountered in the cadmium electroplating process. During the subsequent baking of the probe at a closely controlled temperature, the probe ion current, proportional to hydrogen pressure, is recorded as a function of time. From these data and

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

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[2] 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.

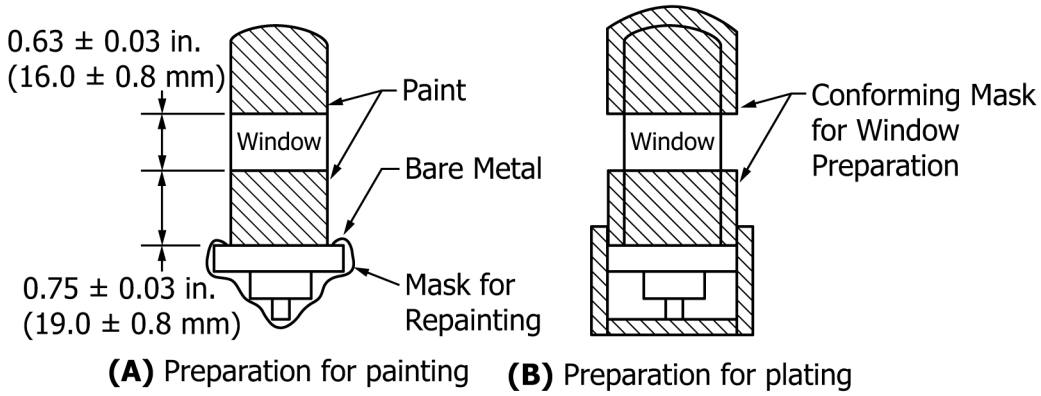

FIG. 1 Probe Configuration

the calibration data of the probe, a number related to the porosity of the electroplated metal relative to hydrogen is obtained.

4.2 During the initial part of the bakeout, hydrogen continues to diffuse through the metal shell of the probe and the ion current increases. Within a short time, however, a maximum current is observed and then falls off as hydrogen is driven out of the system.

4.3 Observations of the ion current-time curve indicate that the slope of the curve has an empirical relationship with failure data on stress rupture specimens such as those in Test Method F519. For this method, *HP* and λ variables (see Section 3) must be empirically correlated with results from the stress rupture specimens. This gives a quick means of measuring ease of baking hydrogen out of cadmium-electroplated parts.

4.4 Before an electroplating test, calibration is accomplished by electrolyzing the probe in a standard solution and baking it to determine *HP* and λ of the unplated steel shell of the probe.

5. Significance and Use

5.1 Hydrogen is evolved during metal electrodeposition in aqueous baths. Some of this hydrogen enters parts during plating. If the absorbed hydrogen is at a level presenting embrittlement hazards to high-strength steel, it is removed by baking parts after plating to expel this hydrogen. However, the lack of plate porosity itself may block hydrogen egress. Thus, it becomes important to know both the relative amount of hydrogen absorbed and the plate porosity.

5.2 This test provides a quantitative control number for cadmium plate porosity that can be used to control a cadmium plating process and the status of cadmium-plated hardware. It can also be used for plating process troubleshooting and research and development to determine the effects on plate porosity by process variables, contaminants, and materials. When used to control a critical process, control numbers for plate porosity must be determined by correlation with stress rupture specimens or other acceptable standards.

5.3 There is no prime standard for plate porosity. For this reason, two ovens must be used, with tests alternated between ovens. Data from the ovens are compared to ensure no equipment change has occurred.

6. Apparatus

6.1 *Hydrogen Detection Instrument*—A system consisting of a control unit, two special ovens, auxiliary heater, recorder, test probes, and associated equipment.

6.2 *Oven*—The oven warms the probe to increase the hydrogen diffusion rate into the probe. Oven parameters are selected by apparatus manufacturer to provide a standard reading for all hydrogen detection instruments.

6.3 *Oven Stopper*—Stopper covering the oven opening. Remove 10 s before inserting the probe.

6.4 *Window*—The window is the unpainted, bare steel portion of the probe, 0.63 in. ± 0.03 in. in height, that is plated in the solution under test. The window is shown in Fig. 1.

6.5 *Abrasive Blast*—Abrasive blast window area in the same way, using the same media, as used for the parts. Probe should be rotated while being blasted to provide uniform surface.

6.6 *Electronic Bakeout Unit*—This heats the probe electrically to remove hydrogen absorbed into the probe after testing. May be part of hydrogen detection instrument.

7. Reagents and Materials

7.1 *Reagents:*

7.1.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

[3] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.