Designation: B 741 – 95 (Reapproved 2000)

Standard Test Method for Porosity In Gold Coatings On Metal Substrates By Paper Electrography [1]

This standard is issued under the fixed designation B 741; 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 equipment and techniques for determining porosity in gold coatings, particularly electrodeposits and clad metals used on electrical contacts.

1.2 This test method is designed to show whether the porosity level is less or greater than some value which by experience is considered by the user to be acceptable for the intended application.

1.3 This test method is suitable for flat surfaces, or with appropriate fixtures, for gently curved surfaces.

1.4 A variety of other porosity testing methods are described in Guide B 765 and in the literature. Detailed critical reviews of porosity testing are also available.[2], [3], [4] Other porosity test methods are B735, B798, B799, and B809.

1.5 This test method is specifically designed for use with gold coatings (>95 % gold) on substrates of copper and nickel and their alloys. Although not covered in this standard, variations of this test method may be found useful in evaluating coatings other than gold.

1.6 The values stated in SI units are to be regarded as standard. The values in parenthesis are for information only.

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 its use.* For a specific precautionary statement, see Note 1.

2. Referenced Documents

2.1 *ASTM Standards:*

B 276 Test Method for Apparent Porosity in Cemented Carbides [5]

B 374 Terminology Relating to Electroplating [6]

B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure [5]

B 542 Terminology Relating to Electrical Contacts and Their Use [5]

B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor [6]

B 765 Guide for Selection of Porosity Tests for Electrodeposits on Related Metallic Coatings [5]

B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography [6]

B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor [6]

B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor ("Flowers-of-Sulfur") [5]

3. Terminology

3.1 *Definitions*—Many terms used in this test method are defined in Terminology B 542 and terms relating to metallic coatings are defined in Terminology B 374.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *decorations*—those reaction products emanating from the pores that provide visual contrast with the paper.

3.2.2 *measurement area (or ''significant surface'')*—the surface that is examined for the presence of porosity. The significant surfaces or measurement areas of the part to be tested shall be indicated on the drawing of the part or by provision of suitably marked samples.

3.2.3 *Discussion*—For specification purposes, the significant surfaces or measurement areas are often defined as those portions of the surface that are essential to the serviceability or function of the part, such as its contact properties, or which can be the source of corrosion products or tarnish films that interfere with the function of the part.

---

[1] This test method is under the jurisdiction of ASTM Committee B02 on Nonferrous Metals and Alloys and is the direct responsibility of Subcommittee B02.11 on Electrical Contact Test Methods.

Current edition approved April 15, 1995. Published June 1995. Originally published as B 741 – 85. Last previous edition B 741 – 90.

[2] For example: Nobel, F. J., Ostrow, B. D., and Thompson, D. W., "Porosity Testing of Gold Deposits," *Plating*, Vol 52, 1965, p. 1001.

[3] Clarke, M., in "Properties of Electrodeposits—Their Measurement and Significance," Edited by Sard, R., Leidheiser, H., and Ogburn, F. *The Electrochemical Society*, Princeton, NJ, 1975.

[4] Krumbein, S. J., "Porosity Testing of Contact Platings," *Proceedings*, Connector and Interconnection Technology Symposium, Oct. 1987, Phila., PA, p. 47.

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

[6] *Annual Book of ASTM Standards*, Vol 02.04.

3.2.4 *metallic coatings*—include platings, claddings, or other metallic layers applied to the substrate. The coating can comprise a single metallic layer or a combination of metallic layers.

3.2.5 *porosity*—the presence of any discontinuity, crack, or hole in the coating that exposes a different underlying metal.

3.2.6 *underplate*—a metallic coating layer between the substrate and the topmost layer or layers. The thickness of an underplate is usually greater than 0.8 µm (30 µin.).

4. Summary of Test Methods

4.1 This test method uses chemically impregnated moistened paper pressed against the specimen surface at controlled pressure.[7] Current is passed from the specimen, which is anodic, to an inert cathode, at a fixed voltage for a specified time. Base metal ions at pore sites migrate to the paper where a reaction occurs. Depending on the reagents employed, this gives either colored products or colorless products that can be made visible by developing the paper in appropriate chemical vapors or solutions.

4.2 This porosity test involves corrosion reactions in which the products delineate defect sites in coatings. Since the chemistry and properties of these products may not resemble those found in natural or service environments, these tests are not recommended for prediction of the electrical performance of contacts unless correlation is first established with service experience.

5. Significance and Use

5.1 Gold coatings are often specified for the contacts of separable electrical connectors and other devices. Electrodeposits are the form of gold that is most used on contacts, although it is also employed as clad metal and as weldments on the contact surface. The intrinsic nobility of gold enables it to resist the formation of insulating oxide films that could interfere with reliable contact operation.

5.2 In order that the nobility of gold be assured, porosity, cracks, and other defects in the coating that expose base metal substrates and underplates must be minimal or absent, except in those cases where it is feasible to use the contacts in structures that shield the surface from the environment or where corrosion inhibiting surface treatments for the deposit are employed. The level of porosity in the coating that may be tolerable depends on the severity of the environment to the underplate or substrate, design factors for the contact device like the force with which it is mated, circuit parameters, and the reliability of contact operation that it is necessary to maintain. Also, when present, the location of pores on the surface is important. If the pores are few in number and are outside of the zone of contact of the mating surfaces, their presence can often be tolerated.

5.3 Methods for determining pores on a contact surface are most suitable if they enable their precise location and numbers to be determined. This test method is well suited to determining pore location. It is also well suited to determine the number of pores per unit area up to a limit where the pore indications begin to overlap to the extent that counting individual indications is not feasible.

5.4 The relationship of porosity levels revealed by particular tests to contact behavior must be made by the user of these tests through practical experience or by judgment. Thus, absence of porosity in the coating may be a requirement for some applications, while a few pores in the contact zone may be acceptable for another.

5.5 The severity of porosity-determining tests may vary from procedures capable of detecting all porosity to procedures that detect only highly porous conditions. The test method in this document is regarded as severe.

5.6 This test method is highly sensitive and is capable of detecting virtually all porosity or other defects in gold coatings that could participate in substrate corrosion reactions. The test is rapid, simple, and inexpensive. It provides good pore delineation in areas where there are several pores. The time for this test is usually on the order of 60 s.

5.7 Since the results are printed on paper, a permanent record of the porosity is obtained.

6. Limitations

6.1 This test is considered destructive in that it reveals the presence of porosity by attacking the base metal areas and by undercutting the gold coating at pore sites or at the edges of the gold coated areas. Any parts exposed to this test shall not be placed in service.

6.2 The paper electrographic procedure is used for gold coatings on substrates of copper or copper alloys with or without a nickel underlayer. This test is faster than the sulfur dioxide, nitric acid vapor, and gel bulk electrographic tests, but it requires that the test specimen be flat or of such shape that a uniform clamping force can be applied.

6.3 This test is intended to be used for quantitative descriptions of porosity (such as number of pores per unit area or per contact) only on coatings that have a pore density sufficiently low that the corrosion sites are well separated and can be readily resolved. As a general guideline this can be achieved for pore densities up to about 100/cm^2. Above this value the tests may still be useful for the qualitative detection and comparisons of porosity.

7. Apparatus

7.1 *Regulated Variable Power Supply* is required. The voltage shall have a ripple of less than 0.1 V at the working voltage and be adjustable from 0 to 5 V with the capability of being set to ±0.2 V. The power supply shall not develop a voltage transient that exceeds the test value by more than 0.1 V. The voltmeter shall have a range of at least 0 to 7 V, capable of readings to 0.2-V resolution. The ammeter shall have a range of at least 0 to 10 mA with 0.5-mA resolution.

7.2 *Press for Clamping Test Specimen and Paper Together* is required. The clamping force shall be adjustable so that clamping pressures on the specimen from 0 to 3.4 MPa (0 to 500 lb/in^2) can be produced. A means of measuring clamping force must be provided. The accuracy of the force measurement must be adequate to assure that the clamping pressure can be measured with an accuracy of ±0.3 MPa (±50 lb/in^2). The

---

[7] Noonan, H. N., “Electrographic Determination of Porosity in Gold Electrodeposits,” *Plating*, Vol 53, 1966, pp. 461–470.

member applying the pressure shall be fabricated from corrosion-resistant metal such as aluminum, stainless steel, or titanium. A pressure member made from these materials shall be lightly abraded periodically to remove the passivating layer that develops with use. An alternative is to plate the member with a coating of gold sufficiently thick and nonporous to eliminate corrosion. The pressure member is connected to the negative terminal of the power supply and the test specimen to the positive. (see Fig. 1)

7.3 *Dye Transfer Paper*— cut in pieces large enough to cover the test specimen.

7.4 *Timer* shall be used to determine the length of the test.

7.5 *Stereomicroscope* having 10X magnification and an illuminator are required for inspection of the electrographic print after the test.

8. Reagents

8.1 Use reagent or "certified" grade chemicals in the preparation of all solutions. In the case of ethanol, reagent grade ethanol, or "specially-denatured" (with methyl and isopropyl alcohols only) may be used.

9. Solutions

NOTE 1—**Precaution:** Observe good laboratory practice in handling all chemicals so as to avoid contact of chemicals with the operator's skin or eyes.

9.1 *Method Procedure A—Two Step Method*:

9.1.1 The electrolyte is 0.01 M with respect to both sodium chloride and sodium carbonate. It is prepared by dissolving 0.59 g of sodium chloride and 1.06 g sodium carbonate in deionized water and adding deionized water to make 1 L.

9.1.2 The developing solution is prepared by first dissolving the dimethylglyoxime in ethyl alcohol to make a 1 % solution. This ethanol solution is then combined with an equal volume of deionized water to which a few drops of ammonia have been added. Pore decorations on paper with this developing solution are red for nickel ions and green-to-brown for copper ions.

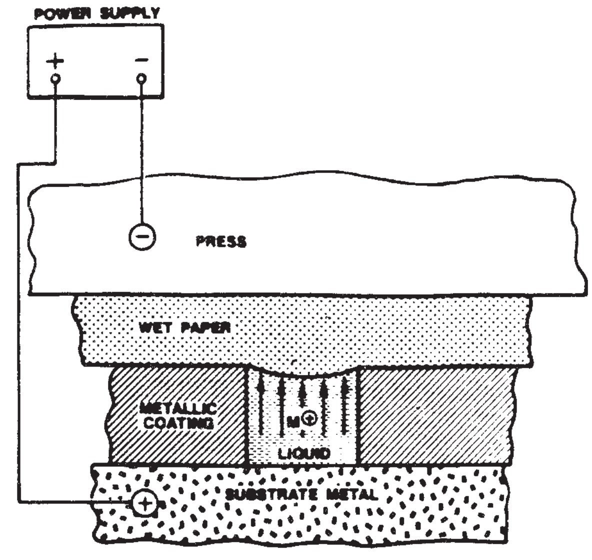

FIG. 1 Schematic Diagram of the Paper-Electrography Test Method

NOTE 2—Caution should be exercised in interpreting the results of this test with certain substrates or underplates which may passivate with time. An example is tin-nickel, a 1:1 intermetallic alloy having a weight composition of approximately 65 % Sn-35 % Ni. This material is reactive in these porosity tests when freshly plated, but becomes passive when exposed to air over a period of days to weeks. The" apparent" porosity of gold at pore sites over such materials may appear to diminish with time, following plating.

9.1.3 An alternative developing solution may be prepared by dissolving 0.25 g of rubeanic acid (dithio oxamide, NH_2-$CSCSNH_2$) in 100 mL of ethanol. The ethanol may be warmed gently to aid in dissolving the solids. Pore decorations on paper treated with this solution are green for copper ions and purple for nickel ions, although the purple may be masked by the green from any copper ions present.

9.2 *Method Procedure B—Single Step Method*:

9.2.1 This method uses a combined indicator-electrolyte solution prepared by adding 20 g/L sodium chloride and 20 g/L of the disodium salt of dimethylglyoxime ($CH_3C{:}NONaC{:}NONaCH_3 \cdot 8H_2O$) to deionized water. A new solution shall be prepared every two weeks or if the solution appears other than colorless, whichever occurs first. The solution shall be stored in a closed container. Pore decorations on paper treated with this solution are green for copper ions and red for nickel ions.

10. Procedure

10.1 Handle specimens as little as possible, and only with tweezers, microscope-lens tissue, or clean, soft cotton gloves. Prior to the test, inspect the samples under 10× magnification for evidence of particulate matter. If present, such particles shall be removed by blowing them off with clean, oil-free air. If this is not successful discard the sample. Then, clean the samples with solvents or solutions that do not contain chlorinated hydrocarbons, CFC's or other known ozone-destroying compounds. The procedure outlined in Note 3 has been found to give satisfactory results for platings with mild to moderate surface contamination.

NOTE 3—Suggested cleaning procedure:

(1) Keep individual contacts separated if there is a possibility of damage to the measurement areas during the various cleaning steps.

(2) Clean samples for 5 min in an ultrasonic cleaner which contains a hot (65–85°C) 2 % aqueous solution of a mildly alkaline (pH 7.5–10) detergent (such as Micro or Sparkleen).

(3) After ultrasonic cleaning, rinse samples under warm running tap water for at least 5 s.

(4) Rinse samples ultrasonically for 2 min in fresh deionized water to remove the last detergent residues.

(5) Immerse in fresh methanol or isopropanol, and ultrasonically "agitate" for at least 30 s in order to remove the water from the samples.

(6) Remove and dry samples until the alcohol has completely evaporated. If an air blast is used as an aid to drying, the air shall be oil free, clean, and dry.

(7) Do not touch measurement area of the samples with bare fingers after cleaning.

(8) Re-inspect samples (under 10× magnification) for particulate matter on the surface. If particulates are found, repeat the cleaning steps. Surface cleanliness is extremely important; contaminants, such as plating salts, organic films, and metal flakes may give erroneous indications of defects, and are unacceptable.

10.2 Soak the dye transfer paper in the electrolyte solution for at least 15 but less than 45 min (preferably 20 to 25 min). Remove excess electrolyte from the paper by blotting with a filter paper. Do not use paper that has been in contact with the electrolyte for more than 45 min, since the emulsion on the paper may be excessively softened. This could cause difficulty later in the procedure.

NOTE 4—A good technique when conducting many tests is to initially immerse three to five strips of paper, then add one strip as one is removed. This allows continuous testing with about 5 min between samples.

10.3 Place the paper emulsion side (glossy side) down on the test specimen and slide it under the clamping member. Connect the clamping member to the negative terminal of the power supply and the test specimen to the positive terminal.

10.4 Adjust the clamping force and the voltage. Values that have been found satisfactory are a clamping force sufficient to produce 1.0 MPa (150 psi) pressure, a voltage of 2 V ± 10 %, and a time of 60 s, ±3 s. (see 7.2 for comments on acceptable pressure accuracy).

10.4.1 If the clamping pressure is too low, background color from the reagents and base metal appearing at pore sites and coating edges may obscure the color decorations. If the pressure is too high, so much electrolyte may be squeezed from the treated paper that few or no decorations will appear in the print. This condition can be detected by testing a specimen on which a scratch through the coating has been made.

10.4.2 The observed current typically falls in the range from 0.05 to 0.5 mA/cm^2 (0.3 to 3.0 mA/in.^{2}) of specimen area. Much lower values indicate an electrical problem such as poor contact due to a passivating layer or contamination. The low current condition produces an inaccurate porosity print. A high-current reading, that is, off scale, indicates a short circuit between the electrodes. This can occur if the paper is out of position. In either event, repeat the test with a new specimen.

10.5 Turn off the power, release the clamp, and carefully peel the paper away from the specimen.

10.6 If Procedure A with the separate electrolyte and developing solutions is in use, dip the paper in the developing solution or wipe with a cloth saturated with developing solution. Then place the paper face down on some *soft* paper toweling and press with a roller to remove the excess developing solution. The paper can then be left to dry face up for about 5 min (heating is usually not necessary with the paper recommended in 7.3). Pore sites will appear as colored spots (pore decorations) on the white paper. The paper may be kept as a permanent record.

10.6.1 If Procedure B is used, no further development is required.

10.7 Alternatively, the paper may be dried in an oven operating between 50 and 70°C. Drying time will vary depending on temperature and the size of the paper. Remove the paper immediately when it is dry; prolonged heating may cause the paper to turn brown.

10.8 Examine the print under direct illumination (at an angle of 75 to 90° to the surface). Either an incandescent lamp or fluorescent ring light may be used.

10.9 If pores are to be counted, duplicate counts shall be made of each, and the two readings averaged. Pore decorations are often counted according to size. A useful sizing technique is to tabulate the pores in accordance with three size ranges. These are (approximately):

10.9.1 A 0.005-in. (0.13 mm) diameter *or less*.

10.9.2 Between 0.005 and 0.015-in. (0.13 and 0.38-mm) diameter.

10.9.3 Greater than 0.015-in. (>0.38-mm) diameter. For sizing purposes, the longest dimension is taken as the diameter. If two or more, generally circular spots, overlap but appear to have separate origins, each is counted as an individual pore and classified in the appropriate size range.

NOTE 5—It is often difficult to determine if decorations on the print which intersect the edge of the gold-coated area originate from defects in the gold or from the base metal normally present beyond the edge of the gold. It is, therefore, suggested that the supplier and user agree upon a procedure for treating such decorations. If the edge of the coated area is not critical in the performance of the part, it may be appropriate to disregard decorations which appear to originate from locations within 0.005 in. (0.13 mm) of the coating edge.

11. Interpretation of Results and Report

11.1 *Report*:

11.1.1 The porosity evaluation shall be determined and reported in accordance with one or more of the options described in Guide B 765.

11.1.2 *Option 1*—The number and size of the pores in the significant area. This is converted to a pore density in terms of number of pores per square centimetre.

11.1.3 *Option 2*—The percentage of total area covered by the pores.

11.1.4 *Option 3*—The area, in square millimetres, of the largest spot on the significant surface.

11.2 *Interpretation of Results*:

11.2.1 In option 1, count individual pores at 10× magnification in the significant area of the coating, as defined by the specification or drawing of the product. In Option 2, compare results with panels shown in Test Method B 276 and Practice B 537, to those supplied as criteria by the purchaser. In Option 3, scan for the largest defect.

11.2.2 *Criteria (Pass-Fail)*—Pass-fail criteria, such as pore count greater than 50/cm^2(Option 1), or an area greater than 1 % (Option 2), or a spot or crack with a total area greater than 2.5 mm^2 (Option 3), should be chosen in conformance to the intended function of the coating and the intended use of the objected coated. Such criteria should be agreed upon between the supplier and purchaser.

12. Precision and Bias

12.1 *Precision*—The precision of this test method is being investigated with gold-plated coupons having a nickel underplate. Round-robin results from a series of four runs in each laboratory with a test of similar severity gave coefficients of variation of less than 20 % in each of three participating laboratories. [8] However, poorer precision was obtained when the results of the different laboratories were compared.

---

[8] Krumbein, S. J., and Holden, C. A., “Porosity Testing of Metallic Coatings,” *Testing of Metallic and Inorganic Coatings*, ASTM STP 947, Harding, W. B. and DiBari, G. A. eds., ASTM, 1987, p. 193.

12.2 *Bias*—The porosity of commercially produced contact platings is a property with potentially large sample-to sample variability.[8] Since there is no acceptable reference material suitable for determining the bias for porosity testing, bias has not been determined.

13. Keywords

13.1 electrography; paper; gold coatings; gold platings; metallic coatings; paper electrography; plating porosity; porosity testing

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).