ASTM INTERNATIONAL

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Standard Test Method for
Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor[1]

This standard is issued under the fixed designation B735; 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 procedures for using nitric acid vapor for determining porosity in gold coatings, greater than 0.6 μm (25 μin.) in thickness, 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 that, by experience, is considered by the user to be acceptable for the intended application.

1.3 A variety of other porosity testing methods are described in Guide B765 and in the literature.[2,3] Other porosity test methods are Test Methods B741, B798, B799, and B809.

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

1.5 *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 become familiar with all hazards including those identified in the appropriate Safety Data Sheet (SDS) for this product/material as provided by the manufacturer, to establish appropriate safety, health, and environmental practices, and determine the applicability of regulatory limitations prior to use. Specific precautions are given in Section 8 and 9.4.*

1.6 *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:*[4]
B374 Terminology Relating to Electroplating
B542 Terminology Relating to Electrical Contacts and Their Use
B741 Test Method for Porosity In Gold Coatings On Metal Substrates By Paper Electrography (Withdrawn 2005)[5]
B765 Guide for Selection of Porosity and Gross Defect Tests for Electrodeposits and Related Metallic Coatings
B798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography
B799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor
B809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor ("Flowers-of-Sulfur")

3. Terminology

3.1 *Definitions*—Many terms used in this test method are defined in Terminology B542 and terms relating to metallic coatings are defined in Terminology B374.

3.2 *Definitions of Terms Specific to This Standard:*
3.2.1 *corrosion products, n*—those reaction products emanating from the pores that protrude from, or are otherwise attached to, the coating surface after a vapor test exposure.

3.2.2 *measurement area (or "significant surface"), n*—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.

3.2.2.1 *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.

3.2.3 *metallic coatings, n*—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.

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

3.2.5 *underplate, n*—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 Method

4.1 This test method employs nitric acid (HNO_3) vapor at low relative humidity. Reaction of the gas mixture with a corrodible base metal at pore sites produces reaction products that appear as discrete spots on the gold surface. Individual spots are counted with the aid of a loupe or low-power stereo microscope.

4.2 This test method is suitable for inlays or claddings containing 75 % or more of gold or for electroplatings containing 95 % or more of gold on substrates of copper, nickel, and their alloys, that are commonly used in electrical contacts.

4.3 The nitric acid vapor test is too severe to be used for gold coatings less than 0.6 μm (25 μin.) in thickness. It is also not suitable for coatings that are less noble than gold or platinum, such as palladium and its alloys, or gold-flashed palladium and its alloys. Gold-flashed is defined as a plated thickness of gold between 3 μin and 5 μin.

4.4 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. Contact surfaces are often curved or irregular in shape, and testing methods should be suitable for them. In addition, the severity of porosity-determining tests may vary. This test method is regarded as severe.

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 judgement. Thus, absence of porosity in the coating may be a requirement for some applications, while a few pores on the critical surfaces may be acceptable for another. Such acceptance (or pass-fail) criteria should be part of the product specification for the particular product or part requiring the porosity test.

5.5 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. In addition, it can be used on contacts having complex geometry such as pin-socket contacts. However, it is preferred that deeply recessed sockets be opened to expose their critical surfaces prior to testing.

5.6 This test method is considered destructive in that it reveals the presence of porosity by contaminating the surface with corrosion products and by undercutting the coating at pore sites or at the boundaries of unplated areas. Any parts exposed to these tests shall not be placed in service.

5.7 This test method 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 $100/cm^2$ or per 100 contacts. Above this value the tests are useful for the qualitative detection and comparisons of porosity.

6. Apparatus

6.1 *Test Chamber,* may be any convenient size glass vessel capable of being sealed with a glass lid, such as a glass desiccator of 9 L to 12 L capacity. The ratio of the air space in the chamber (in cubic centimetres) to the nitric acid surface area (in square centimetres) shall be not greater than 25:1.

6.2 *Specimen Holders or Supports*—Supports or hangers shall be made from glass, polytetrafluoroethylene or other inert materials. It is essential that the holders be so designed, and the specimens so arranged, that the circulation of the vapor is not impeded. Specimens shall be at least 75 mm (3 in.) from the liquid surface and at least 25 mm (1 in.) from the vessel walls. Also, the measurement areas of the specimens shall be at least 12 mm (0.5 in.) from each other.

6.2.1 Do not use a porcelain plate or any other structure that would cover more than 30 % of the liquid surface cross-sectional area. This is to ensure that movement of air and vapor within the vessel will not be restricted during the test.

6.3 *Stereomicroscope,* having a 10× magnification, shall be used for pore counting. In addition a movable source of illumination capable of giving oblique lighting on the specimen surface is also useful.

7. Reagent

7.1 *Nitric Acid,* Reagent Grade Concentrated 70 % ± 2 % HNO_3, sp gr 1.415 to 1.420.

8. Safety Hazards

8.1 Carry out this test method in a chemical fume hood, since the gases that are released, mainly when the reaction vessel is opened at the end of each test, are very corrosive.

8.2 Use caution, however, to ensure that drafts that are often found in fume hoods do not cause significant cooling of the chamber walls, that could lead to a rise in the relative humidity and acceleration of the test (see 9.3). It is often convenient to enclose the reaction vessel in a box with a loose-fitting cover.

8.3 Observe normal precautions in handling corrosive acids. In particular, wear goggles completely enclosing the eyes when handling nitric acid, and make eye wash facilities readily available.

9. Procedure

9.1 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 1 has been found to give satisfactory results for platings with mild to moderate surface contamination.

NOTE 1—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 °C to 85 °C) 2 % aqueous solution of a mildly alkaline (pH 7.5 to 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) Reinspect 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.

NOTE 2—If large areas of exposed non-noble metal are present, masking these areas may be necessary. However, when masking is done with plater's tape, take care not to inhibit the flow of acid vapor to the measurement area.

9.2 The ambient temperature and the temperature of the specimens and solution are 23 °C ± 3 °C at the beginning of the test and maintained throughout the test period.

9.3 The relative humidity in the immediate vicinity of the test chamber shall be no greater than 60 %, [6] although 55 % or below is preferred. If the relative humidity is greater than 60 %, do not run this test.

9.4 Add fresh HNO_3 to the bottom of the *clean and dry* test chamber, and immediately close the cover. After 30 min ± 5 min, load the samples, using suitable fixtures, and replace the cover. The ambient relative humidity shall be no greater than 60 % during both the addition of the HNO_3 and the insertion of the samples.[6] (**Warning**—Do not grease the rim of the desiccator nor its cover. If desired, press a minimum of three strips of pressure sensitive polytetrafluoroethylene tape (adhesive side down) at equal intervals around the desiccator rim.)

9.5 Unless otherwise specified, the exposure time to nitric acid vapor shall be 60 min ± 5 min. An exposure time of 75 min ± 5 min is also commonly used for gold thicknesses in the 2 μm to 2.5 μm (75 μin. to 100 μin.) range. A table of convenient exposure times is given in the appendix.

NOTE 3—Variations in exposure time with thickness are often recommended because pores in thicker coatings are deeper and their average sizes are smaller than those in thinner coatings. The nitric acid medium would therefore take longer to penetrate an average pore in thicker coatings compared to thinner ones. On the other hand, when exposure times are too long, the corrosion products will overlap and impair pore delineation. A detailed discussion of these effects is given in Footnote 3.

9.6 Remove the samples at the end of the test and dry in an oven at 125 °C ± 5 °C for 30 min ± 5 min. Then remove from the oven, and place directly into a desiccator containing active desiccant, and allow to cool to room temperature.

NOTE 4—Discard the HNO_3 in a safe, legally-acceptable manner.

9.7 Leave samples in dessicator until actually ready to start the examination. Then open desiccator slowly, since cooling specimens may create a partial vacuum.

9.8 The examination shall take place within 1.5 h of removal from the oven.

9.9 Count individual pores at 10× magnification using collimated incandescent illumination at an oblique angle below 15°. The pore sites will be delineated by the corrosion products protruding from these sites. These solids may be transparent in the case of gold-plated nickel or nickel underplate, and exercise great care in counting, particularly with rough or curved surfaces.

9.10 Measure and count a corrosion product when at least ¾ of the corrosion product falls within the measurement area. Do not count corrosion products that initiate outside the measurement area but fall within it, and that are irregular in shape, (see Fig. 1).

9.11 Occasionally blisters may form with gold plated on nickel or a nickel underplate. This may occur at pore sites and

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[6] For example see: Holden, C. A., and Luik, R., "Critical Nitric Acid Vapor Test Parameters Affecting the Apparent Porosity of Thin and Selectively Plated Gold," *Plating*, Vol 69, May, 1982, p. 110.