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

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 Material Safety Data Sheet (MSDS) for this product/material as provided by the manufacturer, to establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use.* Specific precautions are given in Section 8 and 9.4.

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[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.).

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

[3] Krumbein, S. J., Porosity Testing of Contact Platings, *Proceedings*, Connectors and Interconnection Technology Symposium, Oct. 1987, p. 47.

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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 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 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 ± 1 % 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