



Designation: B 877 – 96

Standard Test Method for Gross Defects and Mechanical Damage in Metallic Coatings by the Phosphomolybdic Acid (PMA) Method¹

This standard is issued under the fixed designation B 877; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test standard covers equipment and methods for using phosphomolybdic acid (PMA) to detect gross defects and mechanical damage including wear through in metallic coatings of gold, silver, or palladium. These metals comprise the topmost metallic layers over substrates of nickel, copper, or copper alloys.

1.2 Recent reviews of porosity testing, which include those for gross defects, and testing methods can be found in the literature.^{2,3} An ASTM guide to the selection of porosity and gross defect tests for electrodeposits and related metallic coatings is available as Guide B 765. Other related porosity and gross defects test standards are Test Methods B 735, B 741, B 798, B 799, B 809, and B 866.

1.3 The values stated in SI units are the preferred units. Those in parentheses are for information only.

1.4 *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:

- B 374 Terminology Relating to Electroplating⁴
- B 488 Specification for Electrodeposited Coatings of Gold for Engineering Use⁴
- B 542 Terminology Relating to Electrical Contacts and Their Use⁵
- B 679 Specification for Electrodeposited Coatings of Palladium for Engineering Use⁴
- B 689 Specification for Electroplated Engineering Nickel Coatings⁴

B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor⁵

B 741 Test Method for Porosity in Gold Coatings on Metal Substrates by Paper Electrography⁵

B 765 Guide to the Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings⁴

B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography⁵

B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor⁵

B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (“Flowers-of-Sulfur”)⁴

B 866 Test Method for Gross Defects and Mechanical Damage in Metallic Coatings by Polysulfide Immersion⁴

3. Terminology

3.1 *Definitions*—Many terms in this test method are defined in Terminology B 374 or B 542.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *base metal, n*, is any metal other than gold, silver, platinum, palladium, iridium, or rhodium. Typical base metals used as underplates or substrates are copper, nickel, tin, lead, and their alloys.

3.2.2 *defect indications, n*—colored droplets resulting from the reaction between the PMA reagent and the underlying metal.

3.2.3 *gross defects, n*, are those breaks in the coating that expose relatively large areas of underlying metal to the environment. Gross defects include those produced by mechanical damage and wear, as well as as-plated large pores with diameters an order of magnitude greater than intrinsic porosity and networks of microcracks.

NOTE 1—Large pores and microcrack networks indicate serious deviations from acceptable coating practice (dirty substrates and contaminated or out-of-balance plating baths).

3.2.4 *intrinsic porosity, n*—the normal porosity that is present, to some degree, in all commercial thin electrodeposits (precious metal coatings for engineering purposes) that will generally follow an inverse relationship with thickness.

NOTE 2—Intrinsic porosity is due to small deviations from ideal plating and surface preparation conditions. Scanning electron microscope (SEM)

¹ This test method is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10. Current edition approved Oct. 10, 1996. Published December 1996.

² Clarke, M., “Porosity and Porosity Tests,” *Properties of Electrodeposits*, ed. by Sand, Leidheiser, and Ogburn, The Electrochemical Society, 1975, p. 122.

³ Krumbein, S. J., “Porosity Testing of Contact Platings,” *Trans. Connectors and Interconnection Technology Symposium*, Philadelphia, PA, October 1987, p. 47.

⁴ *Annual Book of ASTM Standards*, Vol 02.05.

⁵ *Annual Book of ASTM Standards*, Vol 03.04.

studies have shown the diameter of such pores at the plating surface is 1 to 2 μm so only small areas of underlying metal are exposed to the environment.

3.2.5 *measurement area, n*, that portion or portions of the surface that is examined for the presence of gross defects or mechanical damage and wear through. The measurement area shall be indicated on the drawings of the parts or by the provision of suitably marked samples.

3.2.6 *metallic coatings, n*, include electrodeposits, claddings, or other metallic layers applied to the substrate. The coating can comprise a single metallic layer or a combination of metallic layers (gold over palladium).

3.2.7 *porosity (general), n*, is the presence of any hole, crack, or other defect that exposes the underlying metal to the environment.

3.2.8 *underplate, n*—a metallic coating layer between the substrate and the topmost metallic coating. The thickness of an underplate is usually greater than 1 μm , in contrast to a strike or flash, which is usually thinner.

3.2.9 *wear through, n*—the exposure of underplate or substrate as a direct result of wear. Wear through is an observable phenomenon.

3.2.10 *wear track, n*—a mark that indicates the path along which physical contact has been made during a sliding process (the mating and unmating of an electrical contact).

4. Summary of Test Method

4.1 This test method involves the use of a solution of phosphomolybdic acid (PMA), which is a solid complex of molybdenum trioxide, Mo_2O_3 , and phosphoric acid, H_3PO_4 . In this state, molybdenum is very reactive with many free metals and may be used to detect exposed underplates and substrate metals. The part is exposed briefly to fumes of hydrochloric acid to remove oxides in the defect region. A small drop of the aqueous PMA solution is applied to the spot in question using an applicator. If it contacts base metals from exposed underplate or substrate, the Mo_2O_3 will immediately be reduced to lower oxides, forming the intensely colored, molybdenum blue complex (heteropoly blue).⁶

4.2 This test may not be suitable for some precious metal alloy coatings that contain significant concentrations of non-precious metals (base metals) like nickel or copper. (See 3.2.1.)

4.3 The reagents in this test also react with tin, lead, and tin-lead solder.

5. Significance and Use

5.1 The primary purpose of the PMA test is to determine the presence of mechanical damage, wear through, and other gross defects in the coating. Most metallic coatings are intended to be protective, and the presence of gross defects indicates a serious reduction of such protection.

5.2 The protection afforded by well applied coatings may be diminished by improper handling following plating or as a result of wear or mechanical damage during testing or while in service. The PMA test can serve to indicate the existence of such damage.

5.3 This test is used to detect underplate and substrate metal exposed through normal wear during relative motions (mating of electrical contacts) or through mechanical damage. As such, it is a sensitive pass/fail test and, if properly performed, will rapidly detect wear through to base metals or scratches that enter the base metal layers.

5.4 This test is relatively insensitive to small pores. It is not designed to be a general porosity test and shall not be used as such. The detection of pores will depend upon their sizes and the length of time that the reagent remains a liquid.

5.5 This test cannot distinguish degrees of wear through or whether the wear through is to nickel or copper. Once base metal is exposed, the colored molybdenum complex is formed. While relatively small area defects (compared to the area of the droplet) may be seen at the bottom of the drop as tiny colored regions immediately after applying the PMA, any larger areas of exposed base metal will cause the entire droplet to turn dark instantly.

5.6 The PMA test also detects mechanical damage that exposes underplate and substrate metal. Such damage may occur in any postplating operation or even at the end of the plating operation. It can often occur in assembly operations where plated parts are assembled into larger units by mechanical equipment.

5.7 The PMA test identifies the locations of exposed base metal. The extent and location of these exposed areas may or may not be detrimental to performance. The PMA test is not recommended for predictions of product performance, nor is it intended to simulate field failure mechanisms. For such contact performance evaluations, an environmental test known to simulate actual failure mechanisms should be used.

5.8 The PMA test is primarily intended for the evaluation of individual samples rather than large sample lots, since evaluations are normally carried out one at a time under the microscope (see Section 10).

5.9 This test is destructive. Any parts exposed to the PMA test shall not be placed in service.

6. Apparatus

6.1 In addition to the normal equipment (beakers, weighing balances, funnels, etc.) that are a part of every chemical laboratory.

6.2 *Microscope, Optical, Stereo, 10 to 30 \times* —It is preferred that one eyepiece contain a graduated reticle for measuring the defect location. The reticle shall be calibrated for the magnification at which the microscope is to be used, preferably 10 \times .⁷

6.3 Light source (illuminator) for microscope, incandescent.

6.4 Glass volumetric flask, 10 mL.

6.5 Glass bottle of a stable shape and with glass stopper. The bottle opening shall be 2.5 cm (1 in.) minimum. An example is a 50-mL low-form weighing bottle or a flask-shaped weighing bottle.

6.6 *Applicators* (see 9.2)—Platinum wire, 32 AWG, or disposable glass micropipets, 1 or 0.5 μL size.

⁶ Van Wazer, J. P., *Phosphorous and Its Compounds*, Interscience Publishers, New York, 1961.

⁷ Magnification standards suitable for calibrating optical microscopes may be purchased from U.S. National Institute of Standards and Technology, Office of Standard Reference Materials.