Designation: B 866 – 95 (Reapproved 2003)

Standard Test Method for
Gross Defects and Mechanical Damage in Metallic Coatings by Polysulfide Immersion[1]

This standard is issued under the fixed designation B 866; 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 methods for detecting gross defects and mechanical damage (including wear-through) in metallic coatings where the breaks in the coating penetrate down to a copper or copper alloy substrate.

1.2 This test method is suitable for coatings consisting of single or combined layers of any coating that does not significantly tarnish in an alkaline polysulfide solution. Examples are gold, nickel, tin, tin-lead, and palladium, or their alloys.

1.3 Recent reviews of porosity testing (which include those for gross defects) and testing methods can be found in 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 test standards are Test Methods B 735, B 741, B 798, B 799, and B 809.

1.4 The values stated in SI units are to be regarded as the 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 establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*
B 246 Specification for Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes[4]
B 374 Terminology Relating to Electroplating[5]
B 488 Specification for Electrodeposited Coatings of Gold

for Engineering Uses[5]
B 542 Terminology Relating to Electrical Contacts and Their Use[6]
B 545 Specification for Electrodeposited Coatings of Tin[5]
B 605 Specification for Electrodeposited Coatings of Tin-Nickel Alloy[5]
B 679 Specification for Electrodeposited Coatings of Palladium for Engineering Use[5]
B 689 Specification for Electrodeposited Engineering Nickel Coatings[5]
B 733 Specification for Autocatalytic (Electroless) Nickel-Phosphorus Coatings on Metal[5]
B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor[6]
B 741 Test Method for Porosity in Gold Coatings on Metal Substrates by Paper Electrography[6]
B 765 Guide for Selection of Porosity Tests for Electrodeposits and 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 Terminologies B 374 or B 542.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *defect indications*—black or dark colored products resulting from the reaction between the alkaline polysulfide reagent and exposed copper or copper alloy underlying metal.

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

NOTE 1—Such large pores and microcrack networks indicate serious deviations from acceptable coating practice (as, for example, dirty

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basis-metal substrates and contaminated or out-of-balance plating baths).

3.2.3 *intrinsic porosity*—the "normal" porosity that is present, to some degree, in all commercial thin platings (such as in precious-metal coatings for engineering purposes) and will generally follow an inverse relationship with thickness.

NOTE 2—Intrinsic porosity is due primarily to small deviations from ideal plating and surface preparation conditions. Scanning electron microscope (SEM) studies have shown that the diameter of such pores, at the plating surface, is of the order of micrometres, so that only small areas of underlying metal are exposed to the environment.

3.2.4 *measurement area*—the portion or portions of the surface 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.

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

3.2.6 *porosity (general)*—*in a coating*, the presence of any hole, crack, or other defect that exposes the underlying metal to the environment.

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

3.2.8 *wear-through*—the exposure of underplate or basis metal as a direct result of wear. Wear-through is an observable phenomenon.

3.2.9 *wear track*—a mark that indicates the path along which physical contact had been made during a sliding process (such as the mating and unmating of an electrical contact).

4. Summary of Test Method

4.1 The test samples are immersed in an alkaline polysulfide solution at 74°C (165°F) for 60 s. After rinsing and drying, the samples are examined for dark or discolored areas which indicate exposure of copper or copper alloys to the solution through breaks in the coating.

5. Significance and Use

5.1 The purpose of the alkaline polysulfide immersion 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 alkaline polysulfide test serves to indicate if the damage has extended down to the copper or copper alloy basis metal since it will not detect exposed nickel underplate.

5.3 The alkaline polysulfide test has been specified in several ASTM specifications for tin-plated coatings, namely Specifications B 246 and B 545. This test could also be used to detect gross defects and mechanical damage in other metallic coatings, such as tin-nickel alloy (Specification B 605), nickel (Specification B 689), gold (Specification B 488), palladium

(Specification B 679), and autocatalytic nickel-phosphorous coatings (Specification B 733).

5.4 This test detects mechanical damage that exposes copper underplate and copper basis metal. Such damage may occur in any post-plating operation or even towards the end of the plating operation. It is most often seen to occur in product assembly operations.

5.5 If properly performed, this test will also detect wear-through, provided the wear-through reaches a copper or copper-alloy layer.

5.6 Many types of gross defects are too small to be seen, except at magnifications so high (as in SEM) that a realistic assessment of the measurement area cannot be easily made. Other defects, such as many types of wear-through, provide insufficient contrast with the coating surface. Gross defects tests (as with porosity tests) are, therefore, used to magnify the defect sites by producing visible reaction products in and around the defects.

5.7 The polysulfide solution will react with copper and copper alloys to produce a dark brown or black stain (the defect indications) at the site of the defect. Silver also turns black under the same conditions. The test solution will not react with nickel and is only useful when the presence or absence of copper exposure is a specific requirement.

5.8 The polysulfide immersion test is relatively insensitive to the presence of small pores. It shall not be used as a general porosity test. (Test Method B 809 should be used instead.)

5.9 The extent and location of the gross defects or mechanical damage (revealed by this test) may or may not be detrimental to product performance or service life. Such determinations shall be made by the user of the test through practical experience or judgment.

5.10 The present test can be used on samples of various geometries, such as curved surfaces. It can also be used for selective area coating if allowance is made for tarnish creepage from bare copper alloy areas.

5.11 This test is destructive in that it reveals the presence of gross defects by contaminating the surface with reaction-product films. Any parts exposed to this test shall not be placed in service.

5.12 However, the defect indications on the sample surfaces that result from this test are stable; samples may be retained for reference purposes.

5.13 This test is neither recommended for predictions of product performance nor is it intended to simulate field failure mechanisms. For such product performance evaluations, an environmental test that is known to simulate actual failure mechanisms should be used.

6. Apparatus

6.1 In addition to the normal equipment (beakers, bottles, weighing balances, funnels, and so forth) that are part of every chemical laboratory, the following apparatus are required:

6.1.1 *Microscope*—Optical, stereo, 10 to 30×. It is preferred that one eyepiece contain a graduated reticle for measuring the diameter of tarnish spots. The reticle shall be calibrated for the magnification at which the microscope is to be used, preferably 10×.