Endorsed by American Electroplaters' Society Endorsed by National Association of Metal Finishers

AMERICAN SOCIETY FOR TESTING AND MATERIALS 100 Barr Harbor Dr., West Conshohocken, PA 19428 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method¹

This standard is issued under the fixed designation B 504; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

- 1.1 This test method covers the determination of the thickness of metallic coatings by the coulometric method, also known as the anodic solution or electrochemical stripping method.
- 1.2 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 ISO Standard:

ISO 2177 Metallic Coatings—Measurement of Coating Thickness—Coulometric Method by Anodic Dissolution²

3. Summary of Test Method

- 3.1 The thickness of the coating is determined by measuring the quantity of electricity (coulombs) required to dissolve the coating anodically from a known and accurately defined area.
- 3.2 As commonly practiced, the method employs a small metal cell which is filled with an appropriate electrolyte. The test specimen serves as the bottom of the cell and an insulating gasket between the cell and the specimen defines the test area (about 0.1 cm²). With the test specimen as anode and the cell as cathode, a constant direct current is passed through the cell until the coating has dissolved, at which time a sudden change in voltage occurs.
- 3.3 The thickness of the coating may be calculated from the quantity of electricity used (current multiplied by time), the area, the electrochemical equivalent of the coating metal, the anodic-current efficiency, and the density of the coating. Alternatively, the equipment may be calibrated against standards with known coating thicknesses.

3.4 Commercial instruments using this principle are avail-

able. The method is rapid and versatile, but destructive to the coating. In general, its range is considered to be between 0.75 and 50 μm . Chromium, gold, tin, and other coatings can be measured down to 0.075 μm .

4. Significance and Use

- 4.1 Measurement of the thickness of a coating is essential to assessing its utility and cost.
- 4.2 The coulometric method destroys the coating over a very small (about 0.1 cm²) test area. Therefore its use is limited to applications where a bare spot at the test area is acceptable or the test piece may be destroyed.

5. Factors Affecting the Accuracy of the Method

- 5.1 Composition of Electrolytes—Electrolytes used for coulometric thickness measurements must permit the coating metal to dissolve at a constant anodic-current efficiency (preferably 100%); they must have a negligible spontaneous chemical effect on the coating metal and must so differentiate electrochemically between the coating and the substrate that a suitably sharp and large voltage change occurs at the end point of the test.
- 5.1.1 Electrolytes furnished with commercial instruments may be presumed to meet these requirements; others must be evaluated before use by testing standards having known thicknesses. Appendix X1 lists some electrolytes and coating-substrate combinations that have been used with some instruments.
- 5.2 Current Variation—For coulometric instruments employing the constant-current technique, variation of the current during a test will result in errors. For instruments using a current-time integrator, variation of the current during a test will not result in error unless the current change is such as to displace the anodic current density beyond the range of constant or 100 % anodic-current efficiency.
- 5.3 Area Variation—The accuracy of the thickness measurement will not be better than the accuracy with which the test area is defined or known. Typically, this test area is defined by a flexible, insulating gasket. Area variation is usually minimized by using as large an area as practical and by using a constant pressure device. If excessive pressure is applied to such a gasket, the test area may be altered undesirably.

¹ This test method is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatingsand is the direct responsibility of Subcommittee B08.10on General Test Methods.

Current edition approved Feb. 23, 1990. Published April 1990. Originally published as B 504 - 70. Last previous edition B 504 - 89.

² Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.