



Designation: B1026 – 24

Standard Test Method for Measurement of Thickness of Chromium and Chromium Alloy Deposits by the Coulometric/White Light Interferometry¹

This standard is issued under the fixed designation B1026; 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 the determination of the thickness of metallic coatings by the coulometric method, also known as the anodic solution or electrochemical stripping method, combined with a white light interferometry.

1.2 This test method is particularly applicable to decorative trivalent chromium deposits but can be used for other thin chromium deposits. The range of testing is typically 0.1 μm to 0.5 μm , but it can include thicker deposits.

1.3 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *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 *ISO Standard*:²

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

3. Summary of Test Method

3.1 The thickness of the coating is determined by white light interferometry three-dimensional (3-D) profilometer. The me-

tallic coating is dissolved through the coulometric method using the quantity of electric charge (coulombs) required to dissolve the coating anodically then measuring the thickness using a white light interferometry 3-D profilometer.

3.2 As commonly practiced, the method uses a small metal cell that 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 cm^2). Using the test specimen as an anode and the cell as a cathode, a constant direct current is passed through the cell until the coating has dissolved, at which time a sudden change in voltage occurs. This change in voltage defines when the full layer of the coating has been anodically removed.

3.3 The thickness of the coating may be calculated by placing the specimen on a white light interferometry 3-D profilometer. This thickness is determined by measuring the difference in height from the top of the existing coating to the bottom of the stripped coating.

3.4 Commercial instruments using this principle are available. The method is rapid and versatile but destructive to the coating.

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 cm^2) test area. Therefore, its use is limited to applications in which a bare spot at the test area is acceptable or when the test piece may be destroyed.

5. Factors Affecting the Accuracy of the Test Method

5.1 *Complete Removal of Coating*—The coulometric cell removes a distinct circular area that is easily measured with the white light interferometry 3-D profilometer. Incomplete removal of the coating can affect the accuracy of the measurement.

5.1.1 Electrolytes furnished with commercial instruments may be presumed to meet these requirements; others shall be evaluated before use by testing standards having known

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² Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <https://www.iso.org>.

thicknesses. In [Appendix X1](#), some electrolytes and coating-substrate combinations that have been used with some instruments are listed.

5.2 Substrate Irregularities—Irregularities in the substrate could affect the ability and accuracy of the test method. Specifically, rough or textured base substrates may make it difficult to measure the coating thickness.

5.3 Quantity and Location of Tests—Since the coulometric test method measures a local coating thickness, a single test may not be representative of the coating thickness over the entire significant surface.

6. Calibration of Equipment

6.1 The equipment shall be calibrated by means of standards having known coating thicknesses. If commercial equipment is used, the manufacturer's instructions shall be followed insofar as they are compatible with this test method.

6.2 Calibration of Direct-Reading Instruments—Direct-reading instruments shall be calibrated against standards having known coating thicknesses.

6.3 Thickness Standards—The thickness standards shall consist of the same type of coating and substrate as the specimens to be measured and they shall have an accuracy of $\pm 5\%$ or better.

6.4 Calibration Procedure—The step height or thickness calibration procedure is used to account for disparities between the optical profilometer and the standard.

6.4.1 The standard of known thickness is used as a reference and the step height between the base or uncoated area and the coated layer is measured.

6.4.2 The step height or thickness value shall be compared to the calibration value.

6.4.3 If a correction is applied, then any measurements shall be completed after the correction is made.

6.4.4 Only accept a correction change of 10 % of thickness or less. If a value is greater than 10 %, contact the instrument manufacturer.

7. Procedure

7.1 Procedure for Flat Surfaces (Area Method):

7.1.1 The manufacturer's instructions shall be followed insofar as they are compatible with this test method.

7.1.2 The test surface shall be cleaned of all foreign material that might affect the measurement.

7.1.3 The chromium layer shall be removed using the commercially available coulometric equipment.

7.1.4 Thoroughly rinse the test spot with deionized water and then dry the specimen. Force air dry or blot with a lint-free cloth.

7.1.5 Inspect the area where the coating was removed. Under 10 $\times$ magnification, there should be no traces of the chromium layer still present.

7.1.6 Flat Surfaces—Most flat surfaces will use the standard testing method in [7.1.6.1 – 7.1.6.8](#). In this test method, the instrument uses a large amount of area and averages the step height or thickness for the most accurate reading.

7.1.6.1 Calibrate the instrument step height according to [Section 6](#).

7.1.6.2 Prepare samples for testing by cutting them into proper sample size and paying attention to make the samples as flat (planetary) as possible.

7.1.6.3 Find a region where chromium has been removed and position the area so that two thirds of the screen is stripped area and one third of the area contains chromium ([Fig. 1](#)).

7.1.6.4 Focus using the tip/tilt stage.

7.1.6.5 Adjusting the Tip/Tilt—With the sample loaded onto the stage and focused, level the sample stage to make sure the surface of the sample is as flat as possible relative to the objective. To do this, most 3-D profilometers have a tip/tilt mechanism built into the sample stage, which consists of two adjustment knobs. These knobs lift or lower the stage around a centralized point.

7.1.6.6 Follow the manufacturer's instructions for measuring the sample step height or thickness.

7.1.6.7 Save or record the reading.

7.1.6.8 Between samples, raise the stage to avoid crashing samples into the microscope optics.

7.2 Procedure for Curved Surfaces (Line Method):

7.2.1 In certain cases, a curved surface may cause trouble using the area method for step height or thickness calculation.

NOTE 1—When the surface is curved, the area method may become inaccurate. Proper flatness or focus becomes very difficult and can lead to error and/or poor distribution curves.

7.2.2 If this occurs, you will need to use the line method described in [7.2.2.1 – 7.2.2.8](#). In this method, the instrument gathers data from a straight line across the sample and is not averaging a large area. Therefore, it is recommended to draw a line where the best flatness is obtained and do this three times to average the thickness.

7.2.2.1 Calibrate the instrument step height according to [Section 6](#).

7.2.2.2 Prepare samples for testing by cutting them into proper sample size and paying attention to make the samples as flat (planetary) as possible.

7.2.2.3 Find a region where chromium has been removed and position the area so that two thirds of the screen is stripped area and one third of the area contains chromium ([Fig. 1](#)).

7.2.2.4 Focus using the tip/tilt stage.

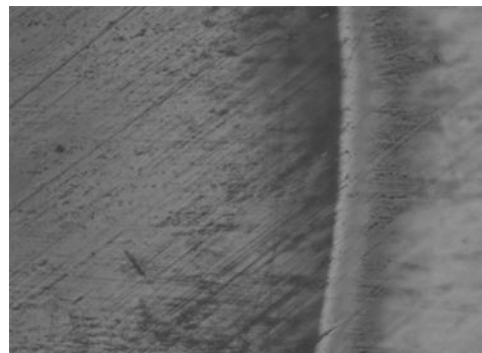


FIG. 1 Find a Region Where Chromium Has Been Removed and Position the Area so that Two Thirds of the Screen is Stripped Area and One Third of the Area Contains Chromium