



Standard Test Method for Simultaneous Thickness and Electrode Potential Determination of Individual Layers in Multilayer Nickel Deposit (STEP Test)¹

This standard is issued under the fixed designation B764; 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 closely estimates the thickness of individual layers of a multilayer nickel electrodeposit and the potential differences between the individual layers while being anodically stripped at constant current density.^{2,3}

1.2 This test method does not cover deposit systems other than multilayer electroplated nickel deposits.

1.3 *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:*⁴

B456 Specification for Electrodeposited Coatings of Copper Plus Nickel Plus Chromium and Nickel Plus Chromium

B504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method

D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 This procedure is a modification of the well-known coulometric method of thickness testing (Test Method **B504**). It is also known as the anodic dissolution or electrochemical stripping method.

3.2 Coulometric thickness testing instruments are based on the anodic dissolution (stripping) of the deposit at constant current, while the time is measured to determine thickness. As commonly practiced, the method employs a small cell that is filled with an appropriate electrolyte, and the test specimen serves as the bottom of the cell. To the bottom of the cell is attached a rubber or plastic gasket whose opening defines the measuring (stripping, anodic) area. If a metallic cell is used, the rubber gasket also electrically insulates the test specimen from the cell. With the specimen as the anode and the cell or agitator tube as the cathode, a constant direct current is passed through the cell until the nickel layer is dissolved. A sudden change in voltage between the electrodes occurs when a different metallic layer starts to dissolve.

3.3 Each different metal or species of the same metal requires a given voltage to keep the current constant while being stripped. As one nickel layer is dissolved away and the next layer becomes exposed, there will be a voltage change (assuming a constant current and difference in the electrochemical characteristics of the two nickel layers). The elapsed time at which this voltage change occurs (relative to the start of the test or previous voltage change) is a measure of the deposit thickness.

3.4 At the same time, the amplitude of the voltage change can be observed. That is, the ease (or difficulty) with which one layer can be dissolved or stripped with reference to another layer can be compared. The lower the voltage needed the more active the metal or the greater the tendency to corrode preferentially to a more noble metal adjacent to it.

3.5 Where the metallic layers are of such a similar nature that change of the stripping voltage is small, there can be problems in detecting this change if the voltage between the deplating cell (cathode) and the sample (anode) is measured. As the sample is dissolved anodically, cathodic processes are occurring on the deplating cell (cathode) surface that can also give rise to voltage changes, due to alterations of the cathode surface, thus obscuring the anode voltage change. This difficulty can be avoided by measuring the potential of the dissolving anodic sample with respect to an unpolarized third electrode (reference) placed in the cell. By recording this

¹ This method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.

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² For discussion of this test, see Harbulak, E. P., "Simultaneous Thickness and Electrochemical Potential Determination of Individual Layers in Multilayer Nickel Deposits," *Plating and Surface Finishing*, Vol 67, No. 2, February 1980, pp. 49–54.

³ U.S. Patent 4,310,389. Assignee: The Chrysler Corp., Highland Park, MI 48203.

⁴ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.