Designation: B 764 – 94 (Reapproved 2003)

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

This standard is issued under the fixed designation B 764; 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 deposit and the electrochemical potential differences between the individual layers while being anodically stripped at constant current density.[2,3]

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 *ASTM Standards:*

B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method[4]

D 1193 Specification for Reagent Water[5]

3. Summary of Test Method

3.1 This procedure is a modification of the well-known coulometric method of thickness testing (Test Method B 504). 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 (electrochemical potential) 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 potential any difference in electrochemical activity between layers is more readily detected.

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[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] *Annual Book of ASTM Standards*, Vol 02.05.

[5] *Annual Book of ASTM Standards*, Vol 11.01.

3.6 The thickness of any specific nickel layer may be calculated from the quantity of electricity used (current multiplied by time), the area dissolved, the electrochemical equivalent of nickel, the anode efficiency, and the density of the nickel layer.

4. Significance and Use

4.1 The ability of a multilayer nickel deposit to enhance corrosion resistance is a function of the electrochemical potential differences between the layers (as measured individually at a fixed current density in a given electrolyte versus a reference electrode) and the thicknesses of the layers. The potential differences must be sufficient to cause the bright nickel or top layer to corrode preferentially and sacrificially with respect to the semibright nickel layer beneath it.

4.2 This test procedure allows the measurement of these potential differences directly on an electroplated part rather than on separate foil specimens in such a way that time determines the thickness of each layer, while the potential difference between nickel layers is an indication of the corrosion resistance of the total nickel deposit.

4.3 The interpretation and evaluation of the results of this test should be by agreement between the purchaser and the manufacturer.

NOTE 1—This test may be used to help predetermine the corrosion resistance of the multilayer nickel coatings applied in production that are subjected to other corrosion media. It should be understood that due to many factors that influence the progress of corrosion during actual use of the part, the performance of different multilayer nickel deposits in the test cannot be taken as an absolute indicator of the relative corrosion resistance of these deposits in service.

5. Apparatus

5.1 *Composition of the Electrolyte*:

Nickel Chloride ($NiCl_2 \cdot 6H_2O$)	300g/L
Sodium Chloride (NaCl)	50g/L
Boric Acid (H_3BO_3)	25g/L
pH	3.0[A]

[A] The pH may be adjusted with diluted hydrochloric acid or sodium hydroxide, as required, and is more critical than the composition of the electrolyte.

Prepared in Purified Water—Type IV or better as specified in Specification D 1193.

5.2 *Constant Current Source*—This should supply a constant current that can be varied between 0 and 50 mA (typical 25 to 35 mA). A current of 30 mA corresponds to a stripping rate of 7.8 µm/min at 100 % current efficiency when used with a gasket providing 0.08 cm^2 stripping area. (This is achieved with the solution stated in 5.1.) Most commercial coulometric thickness testers can be used with slight modification as the current source.

5.3 *Electrolyte Agitation Source*—All commercial coulometric thickness testers incorporate a means to agitate the solution. It is possible to purchase these types of units separately, if so desired, to be used externally in conjunction with other power supplies.

5.4 *Recorder*—Any time-based recorder with an input impedance of at least 1.0 MΩ and capable of running at approximately 0.5 mm/s (3 cm/min) can be used.

5.5 *Deplating Cell*—The cell may be similar in construction to commercially available deplating cells. It is usually a cup-shaped cell of either 316 stainless steel, Monel,[6] or plastic that engages a round rubber or plastic gasket to the work piece or sample. The opening through the cell and gasket allows contact of the electrolyte to the test specimen and defines the stripping area.

NOTE 2—A deplating cell could be constructed of plastic using a cylindrical stainless steel or Monel sheet cathode located in the larger upper area of the cup. The advantages of such a cell are the prevention of whisker growth and the choking off of the small bore opening, and the ease of cathode removal for cleaning or replacement.

5.6 *Reference Electrode Assembly*—The general configuration of one commercially available measuring system is shown in Fig. 1. The assembly is a "T"-shaped housing containing the reference electrode. The housing assembly includes an upper connector providing a port for the connection to the agitator pump. The upper position also contains a threaded contact to the reference electrode. The lower portion of the assembly contains an adjustable glass or plastic tube which surrounds the reference electrode so that when inserted into the deplating cell, the reference electrode is immersed in the electrolyte. The tip of the reference electrode should extend into the glass tip so that the distance between the tip of the electrode and the bottom of the glass agitator tube is approximately 5.0 mm.

5.7 *Reference Electrode*—Either silver or platinum wire of approximately 1.5 mm in diameter can be used. Silver is probably the better choice due to its ability to form a silver-silver chloride electrode when used in a chloride containing electrolyte (see Appendix X1).

NOTE 3—It is necessary to condition the silver electrode before using in order to form the silver-silver chloride surface. This is easily done by anodically treating approximately a 75-mm length of wire in an 1 *N* hydrochloric acid solution for 10 to 15 s using a 34.7-mA anodic current. This will form a gray film on the wire which should always be present. Once the gray film is formed, it is not necessary to repeat the conditioning treatment unless the film has been removed. It may be advisable, however, to recondition the electrode after a prolonged period of inactivity or when the electrode has been allowed to remain dry for an extended period of time. Drying off the electrode should be avoided by immersion in either the hydrochloric acid conditioning solution, the step test solution, or distilled water when not in use.

NOTE 4—A ceramic junction reference electrode that does not require conditioning is available commercially.

5.8 *Millivolt Meter (optional)*—When using a sensitive and well-calibrated recorder, a millivolt meter is not necessary. If one is desired, however, any sensitive, high-input impedance meter can be used. A standard pH meter with a millivolt setting

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[6] Monel is a trademark for a large group of corrosion-resistant alloys of predominantly nickel and copper. Available from Huntington Alloy Products Div., The International Nickel Co., Inc., Huntington, WV 25720.

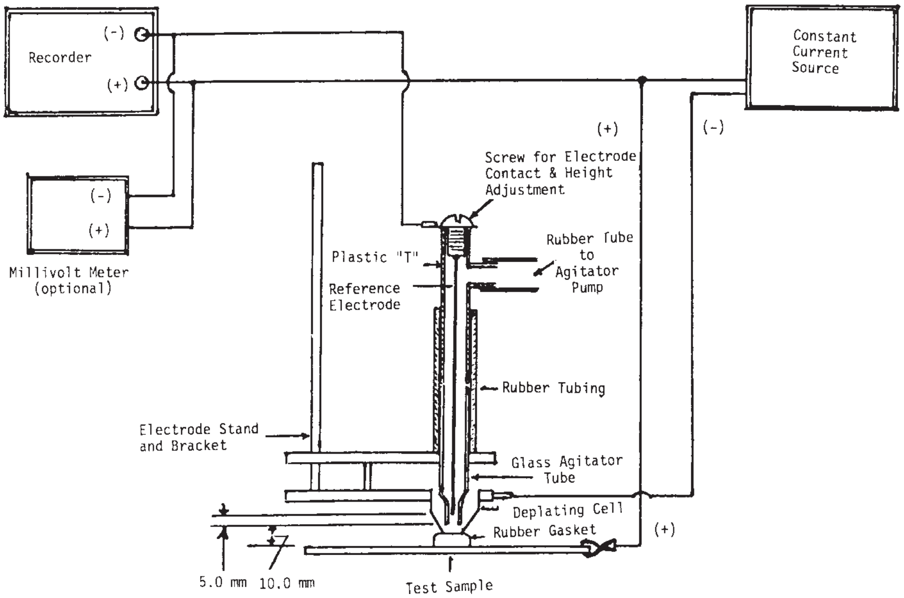


FIG. 1 T-Shaped Reference Electrode Assembly

would be satisfactory. The meter should have a range from 0 to 2000 mV. If a millivolt meter is used which has low-output impedance facilities, it can be used to drive the recorder and will serve as a buffer amplifier. Most laboratory pH meters have such output terminals (see Fig. 2).

6. Procedure

6.1 Set up equipment as recommended by the manufacturer or as shown in Fig. 1. If necessary, turn on the recorder and the millivolt meter and allow them to warm up.

6.2 If chromium is present on the nickel surface, remove it with concentrated hydrochloric acid. Make sure the nickel surface is clean. Rinse well and dry off the surface.

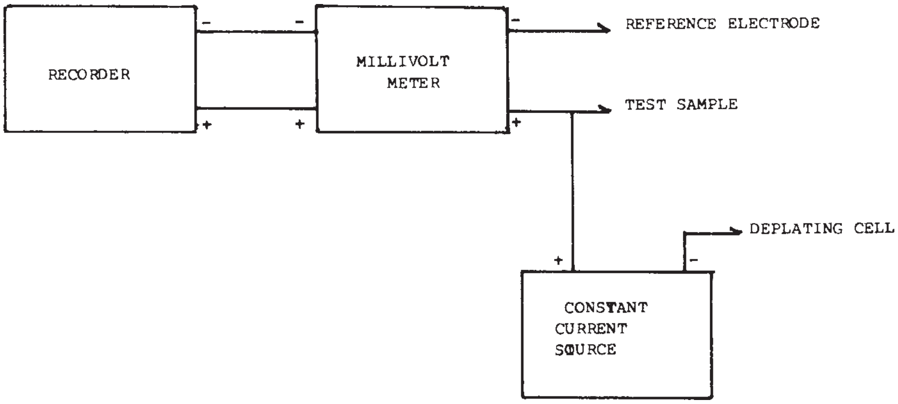
NOTE 5—Chromium can be removed by using the deplating cell as is done on many commercial coulometric testers. If this is done, secure the cell and gasket to the test piece as in 6.3 and 6.4 but do not insert the electrode assembly. Fill the cell with a common test stripping solution for chromium (Test Method B 504) and hook up only the cell and test piece to the power supply. Apply the current until all the chromium has been removed. A dense blanket of bubbles on the surface of the sample indicates that all the chromium is removed. Remove the stripping solution from the cell without moving or disturbing the seal of the gasket to the test surface. Wash the cell three times with purified water (Type IV or better as specified in Specification D 1193) and once with the step test solution. Proceed to 6.5.

6.3 Position the test specimen in a secure horizontal position so that the chromium-stripped nickel surface is directly beneath the cell gasket.

6.4 Lower the deplating cell assembly; secure by sealing the gasket to the nickel surface. A flat test area of approximately 10 mm in diameter is desirable but not required. The criterion is that there be no leakage of the electrolyte. If leakage does occur, discontinue test and start a new one.

6.5 Fill the deplating cell to the appropriate level with the step test solution making sure that no air is trapped within the solution.

6.6 Lower the reference electrode assembly into the deplating cell, if necessary.



NOTE 1—The millivolt meter should have a high-input impedence and a low-output impedence. Typical pH meters have an input impedence of 1014 Ω. Digital meters should have an input impedence of at least 10^7Ω.

FIG. 2 Alternative Circuit Using a High-Impedence Millivolt Meter as a Buffer Amplifier