

Standard Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Electrodeposited Nickel Coatings on Magnetic and Nonmagnetic Substrates¹

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

1. Scope

- 1.1 This test method covers the use of magnetic instruments for the nondestructive measurement of the thickness of an electrodeposited nickel coating on either a magnetic or non-magnetic substrate. It is intended to supplement manufacturers' instructions for the operation of the instruments and is not intended to replace them.
- 1.2 These instruments measure either the magnetic attraction between a magnet and the coating-substrate combination (categorized as "magnetic pull-off"), or the change in magnetic flux density within the probe (categorized as "electronic").
- 1.3 For this test method, there are two types of coatingsubstrate combinations that can be encountered: Type A, nickel coatings on a magnetic substrate, and Type B, nickel coatings on a nonmagnetic substrate.
- 1.4 The effective measuring ranges of instruments using the principle of magnetic attraction are up to 50 μ m (2 mils) for Type A coatings, and up to 25 μ m (1 mil) for Type B coatings. For gages based on change in magnetic flux density principles, the effective ranges are much greater, and measurements up to 1 mm (40 mils) or more, can be made on both types of coatings.
- 1.5 Measurements made in accordance with this test method will be in compliance with the requirements of ISO Standard 2361 as printed in 1982.
- 1.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.
- 1.7 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 appro-

priate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

B487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of Cross Section

B499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals

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

B748 Test Method for Measurement of Thickness of Metallic Coatings by Measurement of Cross Section with a Scanning Electron Microscope

2.2 ISO International Standard:

ISO 2361 Electrodeposited Nickel Coatings on Magnetic and Nonmagnetic Substrates—Measurement of Coating Thickness—Magnetic Method³

3. Terminology

- 3.1 Definitions of Terms Specific to This Standard:
- 3.1.1 *accuracy, n*—the measure of the magnitude of error between the result of a measurement and the true thickness of the item being measured.
- 3.1.2 *adjustment, n*—the physical act of aligning an instrument's thickness readings to match those of a known thickness sample (removal of bias), in order to improve the accuracy of the instrument on a specific surface or within a specific portion of its measurement range. An adjustment will affect the outcome of subsequent readings.

¹ This test 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 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.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.



- 3.1.3 *calibration, n*—the high-level, controlled and documented process of obtaining measurements on traceable calibration standards over the full operating range of the instrument, then making the necessary instrument adjustments (as required) to correct any out-of-tolerance conditions.
- 3.1.3.1 *Discussion*—Calibration of coating thickness instruments is performed by the equipment manufacturer, an authorized agent, or by an authorized, trained calibration laboratory in a controlled environment using a documented process. The outcome of the calibration process is to restore/realign the instrument to meet/exceed the manufacturer's stated accuracy.
- 3.1.4 *reference standard*, *n*—a specimen of known thickness used to verify the accuracy of a coating thickness measuring instrument.
- 3.1.5 *verification of accuracy, n*—obtaining measurements on a reference standard prior to instrument use for the purpose of determining the ability of the coating thickness instrument to produce reliable values, compared to the combined instrument manufacturer's stated accuracy and the stated accuracy of the reference standard.

4. Summary of Test Method

- 4.1 Magnetic pull-off instruments are mechanical instruments that measure the force required to pull a permanent magnet from magnetic material. The magnetic force of attraction to the magnetic coating or coating-substrate combination is opposed by a spring or coil. Tension is applied to the spring/coil until the magnetic attraction to the material is overcome. The instrument must be placed directly on the coated surface to obtain a measurement. The force holding the permanent magnet to the magnetic material is inversely proportional to the thickness of the coating layer(s) between the magnet and the magnetic material. For example, a thin nickel layer applied to a nonmagnetic substrate will require less spring tension to pull the magnet off than will a thicker nickel layer, since the thinner coating has weaker magnetic strength.
- 4.2 Electronic instruments measure a change in magnetic flux density within the probe to produce a coating thickness measurement. The instrument probe must be placed directly (in a perpendicular position) on the coated surface to obtain a measurement. These instruments determine the effect on the magnetic field generated by the probe due to the proximity to the substrate.

5. Significance and Use

- 5.1 The thickness of a coating is often critical to its performance. This magnetic method is suitable for measuring nondestructively the thickness of some nickel coatings and for specification acceptance.
- 5.2 This method requires that the magnetic properties of the coating and its substrate be the same as those of the reference standards used for the calibration adjustment of the instrument.
- 5.3 This method should not be used to determine the thickness of autocatalytically deposited nickel-phosphorus alloys containing more than 8 % phosphorus on steel. Those coatings are sufficiently nonmagnetic for Test Method B499 to

be suitable for that determination, as long as the measurement is made prior to any heat treatment.

6. Apparatus

- 6.1 Coating Thickness Instrument, based on magnetic principles, commercially available, suitable to measure coating thickness accurately.
- 6.2 Coating Thickness Standards , with assigned values traceable to a National Metrology Institution. They may be coated or plated steel plates, or may be foils or shims of flat, non-metallic sheet (typically polyester).

7. Calibration and Standardization

- 7.1 Calibration of coating thickness instruments is performed by the equipment manufacturer, an authorized agent, or by an authorized, trained calibration laboratory in a controlled environment using a documented process. A Certificate of Calibration showing traceability to a National Metrology Institution can be issued. There is no standard time interval for re-calibration, nor is one absolutely required, but a calibration interval can be established based on experience and the work environment. A one-year calibration interval is a typical frequency suggested by many instrument manufacturers.
- 7.2 Before use, each instrument's calibration accuracy shall be verified in accordance with the instructions of the manufacturer, employing suitable thickness standards and, if necessary, any deficiencies found shall be corrected.
- 7.3 During use, calibration accuracy shall be verified at frequent intervals, at least once a day. Attention shall be given to the factors listed in Section 8 and to the procedures described in Section 9.
- 7.4 Reference standards shall be coated standards obtained by electroplating nickel adherently onto a substrate. The coating thickness of the reference standards shall bracket the user's highest and lowest coating thickness measurement requirement.
- 7.5 The substrate and the coating of the standard shall have the same magnetic properties as those of the test specimen (see 8.2, 8.3, 8.11 and 8.12).
- 7.5.1 To assure the similarity of the magnetic properties of the nickel deposit and for Type A coatings on steel substrate, reference standards shall be produced and measured by another suitable test method, such as cross sectioning or the coulometric test method from a specimen produced under identical conditions as the test specimen to be measured. To confirm the similarity of the magnetic properties of the substrate to those of the standards, a comparison of the readings obtained with the bare basis metal of the standard to that of the test specimen is recommended.
- 7.5.2 In the same manner, the similarity of the magnetic properties of the coating of the test specimen to that of the standard can be established by verifying with the cross sectioning (Test Methods B487 or B748) or coulometric (Test Method B504) methods that the thickness reading obtained on the test specimen by means of the properly adjusted instrument corresponds to the actual thickness determined by one or both of the above methods.

- 7.6 Where indicated, the accuracy of the instrument should be checked by rotating the probe in increments of 90° (see 8.7 and 8.8).
- 7.7 For Type A coatings, the basis metal thickness for the test and the calibration adjustment shall be the same if the critical thickness, defined in 8.3, is not exceeded. When possible, back up the basis metal of the standard, or the test specimen, with a sufficient thickness of similar material to make the readings independent of the basis metal thickness.
- 7.8 If the curvature of the coating to be measured is such as to preclude calibration adjustment on a flat surface, the curvature of the coated standard shall be the same as that of the test specimen.

8. Factors Affecting the Measuring Accuracy

- 8.1 Coating Thickness—Inherent in the method is a measuring uncertainty that, for thin coatings, is constant and independent of the coating thickness; for thicknesses greater than about $50 \mu m$ (2 mils), this uncertainty is proportional to the coating thickness.
- 8.2 Magnetic Properties of the Basis Metal (Type A coatings only)—Magnetic thickness measurements are affected by variations in the magnetic properties of the basis metal. For practical purposes, magnetic variations in low-carbon steel can often be considered to be insignificant. To avoid the influences of severe or localized heat treatments and cold working, the instrument should be adjusted using a reference standard having a basis metal with the same magnetic properties as that of the test specimen or, preferably and if available, with a sample of the part to be tested before application of the coating.
- 8.3 Basis Metal Thickness (Type A coatings only)—For each instrument, there is a critical thickness of the basis metal above which the measurements will not be affected by an increase in that thickness. Since it depends on the instrument probe (Note 1) and the nature of the basis metal, its value should be determined experimentally, if it is not supplied by the manufacturer.
 - Note 1—The term "instrument probe" also includes the term "magnet."
- 8.4 Edge Effect—The method is sensitive to abrupt changes in the surface contour of the test specimen. Therefore, measurements made too near an edge or inside corner will not be valid, unless the instrument is specially adjusted for such a measurement. This also applies to measurements made on geometrically limited areas, such as narrow conductors on printed circuit boards.
- 8.5 Curvature—Measurements are affected by the curvature of the test specimen. The influence of curvature varies considerably with the make and type of instrument, but always becomes more pronounced as the radius of curvature decreases. Instruments with two-pole probes may also produce different readings, depending on whether the poles are aligned in planes parallel or perpendicular to the axis of a cylindrical surface. A similar effect can occur with a single-pole probe, if the tip is unevenly worn. Measurements made on curved test specimens may not, therefore, be valid unless the instrument is specifically adjusted for such measurements.

- 8.6 Surface Roughness:
- 8.6.1 Measurements are influenced by the surface topography of the substrate and the coating, and a rough or scratched surface will give individual instrument readings that all vary from point to point. In this case, it is necessary to make many readings at different positions to obtain an average value that is representative of the mean coating thickness.
- 8.6.2 If the basis metal is magnetic and rough, it may also be necessary to check, and adjust if necessary, the zero of the instrument at several positions on a sample of the uncoated rough substrate.
- 8.6.3 If the roughness of the substrate surface is small, relative to the coating thickness, its effect will probably be negligible.
- 8.7 Direction of Mechanical Working of the Basis Metal (Type A coatings only)— Measurements made by an instrument having a two-pole probe or an unevenly worn single-pole probe may be influenced by the direction in which the magnetic basis metal has been subjected to mechanical working, such as rolling. The reading may change with the orientation of the probe on the surface.
- 8.8 Residual Magnetism (Type A coatings only)—Residual magnetism in the basis metal affects the measurements made by instruments that employ a stationary magnetic field. Its influence on measurements made by reluctance instruments employing an alternating magnetic field is much smaller.
- 8.9 Stray Magnetic Field—Strong stray magnetic fields, such as those produced by various types of electrical equipment, can seriously interfere with the operation of instruments based on magnetic principles.
- 8.10 Foreign Particles—The probes of magnetic instruments of all types must make physical contact with the test surface and are, therefore, sensitive to foreign material that prevents intimate contact between the probe and the coating surface. Both the test surface and the instrument probe should be kept free of foreign material.
- 8.11 Magnetic Properties of the Coating— Magnetic thickness measurements are affected by variations in the magnetic properties of the coating. These properties depend on the conditions under which the deposit is produced, the type and composition of the coating, and its stress. The magnetic properties of multiple-layer nickel coatings will also depend on the relative thickness of each of the layers.
- 8.11.1 A heat treatment at $400\,^{\circ}\text{C}$ (750 $^{\circ}\text{F}$) for 30 min will equalize the magnetic permeability of dull (Watts) nickel coatings of the same composition. Bright nickel deposits may or may not have the same magnetic properties after heat treatment.
- 8.12 *Nickel Coatings on the Back of the Substrate (Type B coatings only)* Nickel coatings on the back of the substrate can affect the measurements, depending on the thickness of the substrate.
- 8.13 *Pressure*—Instrument readings are sensitive to the pressure with which the probe is applied to the test specimen. No deformation of the coating or probe should be allowed. Errors that sometimes are encountered with the use of manual

probes can be avoided by employing spring-loaded probes that exert a relatively constant pressure.

8.14 *Probe Orientation*—Instrument readings may be sensitive to the orientation of the magnet in relation to the field of gravity of the earth. Thus, the operation of an instrument in a horizontal or upside-down position may require a correction factor, or may be impossible.

9. Procedure

- 9.1 Operate each instrument in accordance with the instructions of the manufacturer. Give appropriate attention to the factors listed in Section 8.
- 9.2 Verify the accuracy of the instrument at the test site each time the instrument is put into service, and at regular intervals during use, to assure proper performance.
 - 9.3 Observe the following precautions:
- 9.3.1 Basis Metal Thickness (Type A coatings only)—Check whether the basis metal thickness exceeds the critical thickness. If not, either use the back-up method of 7.5, or make sure that the calibration adjustment has been made on a standard having the same thickness and magnetic properties as the test specimen.
- 9.3.2 *Edge Effects*—Do not make readings close to an edge, hole, inside corner, etc., of a specimen, unless the validity of the calibration adjustment for such a measurement has been demonstrated
- 9.3.3 *Curvature*—Do not make readings on a curved surface of a specimen, unless the validity of the calibration adjustment for such a measurement has been demonstrated.
 - 9.3.4 Number of Readings:
- 9.3.4.1 For each measurement, make at least 3 readings, removing the probe after each reading, and average the readings. If any 2 of the readings differ from each other by more than 5 % of the average reading or 2 μm (0.08 mil), whichever is the greater, then the measurement shall be discarded and repeated.
- 9.3.4.2 The substrate or coating, or both, may be too rough to meet this criterion. In such a case it may be possible to obtain a valid measurement by averaging a number of readings. To be valid under this test method, the validity of such a procedure must be demonstrated (see Appendix X1).
- 9.3.4.3 Magnetic pull-off instruments are sensitive to vibrations, and readings that are obviously erroneous should be rejected.
- 9.3.5 Direction of Mechanical Working (Type A coatings only)—If the direction of mechanical working has a pronounced effect on the reading, make the measurement on the test specimen with the probe in the same orientation as that used during the calibration adjustment. If this is impossible, make four measurements at various orientations by rotating the probe in increments of 90°.
- 9.3.6 Residual Magnetism (Type A coatings only)—When residual magnetism is present in the basis metal, it is necessary, when using instruments employing a stationary magnetic field, to make measurements in two orientations differing by 180°. It may also be necessary to demagnetize the test specimen to get valid results (see 8.7 and 8.8).

- 9.3.7 Surface Cleanliness—Before making measurements, clean any foreign matter such as dirt, grease, and corrosion products from the surface without removing any coating material. When making measurements avoid any areas having visible defects that are difficult to remove, such as welding or soldering flux, acid spots, dross, or oxide.
- 9.3.8 *Techniques*—The readings obtained may depend on the technique of the operator. For example, the pressure applied to a probe or the rate of applying a balancing force to a magnet will vary from one individual to another. Such effects can be reduced or minimized either by having the instrument adjusted by the same operator who will make the measurement, or by using constant pressure probes.
 - 9.3.9 Positioning of the Probe:
- 9.3.9.1 In general, place the instrument probe perpendicular to the specimen surface at the point of measurement. For some magnetic pull-off instruments this is essential. With some instruments, however, it is desirable to tilt the probe slightly and select the angle of inclination giving the minimum reading.
- 9.3.9.2 If, on a smooth surface, the readings obtained vary substantially with the angle of inclination, it is probable that the probe is worn and needs to be replaced. If a magnetic pull-off instrument is to be used in a position other than for what it was designed, such as upside-down, apply a correction factor for that position as per the manufacturer's instructions.

10. Report

- 10.1 The report should include the following information:
- 10.1.1 A reference to this standard,
- 10.1.2 Type of instrument used including manufacturer, model number, principle of operation, and date of calibration,
 - 10.1.3 Size and description of test specimen,
- 10.1.4 Whether special jigs were used for accuracy verification and any calibration adjustment,
- 10.1.5 Type of coating thickness standard and/or reference standard and the method used,
- 10.1.6 The number of measurements taken and the value of each measurement,
 - 10.1.7 Operator identification, and
 - 10.1.8 Date.

11. Precision and Bias

- 11.1 The equipment and its operation shall be such that the coating thickness can be determined with an uncertainty of less than 10% at 95% confidence level.
- 11.2 Although an uncertainty of less than 10 % may be achieved consistently for a great number of applications, the uncertainty may be greater when the coating thickness is less than 25 μ m (1 mil).
- 11.3 Instruments suitable for compliance with 9.1 are available commercially. For many coating systems, the instruments are capable of making measurements with an uncertainty of less than 5 % at 95 % confidence level.
- 11.4 The measurement bias is the discrepancy remaining between the measured thickness and the true thickness if all random errors are eliminated. It is, therefore, no greater than,



and attributable to (1) the calibration error of the instrument and (2) the quality of the reference standard used to adjust the instrument.

11.5 The precision is being determined by round-robin testing.

12. Keywords

12.1 coating thickness; magnetic method; nickel coatings; nondestructive thickness; thickness; thickness testing

APPENDIX

(Nonmandatory Information)

X1. MEASUREMENTS ON ROUGH SURFACES

X1.1 Measurements on rough surfaces are subject to random errors associated with the position of the instrument probe relative to the peaks and valleys of the rough surface. These random errors increase with surface roughness, but can be reduced by averaging 10 or more readings.

X1.2 Roughness can also introduce a bias (systematic error) because the probe seldom, if ever, rests at the bottom of a

valley; and the magnetic field in the neighborhood of the probe differs from that at a smooth surface. In the case of a rough substrate, the valleys are filled with coating material, but when the instrument is adjusted with a foil, the foil rests on the peaks of the substrate. A bias can be corrected for if the magnitude of the bias can be determined by microscopical or other measurements.

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