

Designation: B 530 - 96 - 02

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

This standard is issued under the fixed designation B 530; 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 use of magnetic instruments for the nondestructive measurement of the thickness of an electrodeposited nickel coating on either a magnetic or nonmagnetic—substrate. For autoeatalytic nickel coating, see Section 7. substrate.
- 1.2 These instruments measure either the magnetic attraction between a magnet and the coating-substrate combination, or the reluctance of a magnetic flux path passing through the coating and the basis metal.
- 1.3 For this test method, there are two types of coating-substrate 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 $50 \,\mu m$ for Type A coatings, and up to $25 \,\mu m$ for Type B coatings. For reluctance gages, the effective ranges are much greater, and measurements up to $1 \,m m$ 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 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 499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals²

2.2 ISO International Standard:

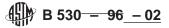
ISO 2361 Electrodeposited Nickel Coatings on Magnetic and Nonmagnetic Substrates—Measurement of Coating Thickness—Magnetic Method²

Annual Book of ASTM

² Available from American National Standards, Vol 02.05. Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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

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3. Significance and Use

- 3.1 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.
- 3.2 This method requires that the magnetic properties of the coating and its substrate be the same as those of the calibration standards.

4. Factors Affecting the Measuring Accuracy

- 4.1 *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 µm, this uncertainty is proportional to the coating thickness.
- 4.2 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 calibrated using a calibration 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.
- 4.3 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."

- 4.4 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 calibrated for such a measurement. This also applies to measurements made on geometrically limited areas, such as narrow conductors on printed circuit boards.
- 4.5 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 calibrated for such measurements.
 - 4.6 Surface Roughness:
- 4.6.1 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.
- 4.6.2 If the basis metal is magnetic and rough, it may also be necessary to check the zero of the instrument at several positions on a sample of the uncoated rough substrate.
 - 4.6.3 If the roughness of the substrate surface is small, relative to the coating thickness, its effect will probably be negligible.
- 4.7 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.
- 4.8 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.
- 4.9 *Stray Magnetic Field*—Strong stray magnetic fields, such as those produced by various types of electrical equipment, can seriously interfere with the operation of magnetic thickness instruments.
- 4.10 *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.
- 4.11 *Magnetic Properties of the Coating* Magnetic thickness measurements are affected by variations in the magnetic properties to 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.
- 4.11.1 A heat treatment at 400°C 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.
- 4.12 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.
- 4.13 *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.
 - 4.14 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 different calibration, or may be impossible.

5. Calibration of Instruments

- 5.1 Before use, each instrument shall be calibrated in accordance with the instructions of the manufacturer, employing suitable thickness standards. During use, the calibration shall be checked at regular intervals, at least once a day. Attention shall be given to the factors listed in Section 4 and to the procedures in Section 6.
- 5.2 The calibration standards shall be coated standards obtained by electroplating nickel adherently onto a substrate. The coating thickness of the calibration standards shall bracket the user's highest and lowest coating thickness measurement requirement.
- 5.3 The substrate and the coating of the standard shall have the same magnetic properties as those of the test specimen (see 4.2, 4.3, 4.12 and section 4.12.1).
- 5.3.1 To assure the similarity of the magnetic properties of the nickel deposit and for Type A coatings the steel substrate, calibration standards shall be produced by another suitable test method, such as cross sectioning or the—C_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.
- 5.3.2 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 coulometric or microscopical method that the thickness reading obtained on the test specimen by means of the properly calibrated instrument corresponds to the actual thickness determined by one or both of the above methods.
- 5.4 Where indicated, the calibration of the instrument should be checked by rotating the probe in increments of 90° (see 4.7 and 4.8)
- 5.5 For Type A coatings, the basis metal thickness for the test and the calibration shall be the same if the critical thickness, defined in 4.3, 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.
- 5.6 If the curvature of the coating to be measured is such as to preclude calibration on a flat surface, the curvature of the coated standard shall be the same as that of the test specimen.

6. Procedure

- 6.1 Operate each instrument in accordance with the instructions of the manufacturer. Give appropriate attention to the factors listed in Section 4.
- 6.2 Check the calibration 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.
 - 6.3 Observe the following precautions:
- 6.3.1 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 5.5, or make sure that the calibration has been made on a standard having the same thickness and magnetic properties as the test specimen.
- 6.3.2 Edge Effects—Do not make readings close to an edge, hole, inside corner, etc., of a specimen, unless the validity of the calibration for such a measurement has been demonstrated.
- 6.3.3 Curvature—Do not make readings on a curved surface of a specimen, unless the validity of the calibration for such a measurement has been demonstrated.
 - 6.3.4 Number of Readings:
- 6.3.4.1 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 μ m, whichever is the greater, then the measurement shall be discarded and repeated.
- 6.3.4.2 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).
- 6.3.4.3 Instruments of the attractive force type are sensitive to vibrations, and readings that are obviously erroneous should be rejected.
- 6.3.5 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 calibration. If this is impossible, make four measurements at various orientations by rotating the probe in increments of 90°.
- 6.3.6 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 4.7 and 4.8).
- 6.3.7 *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.

- 6.3.8 *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 calibrated by the same operator who will make the measurement, or by using constant pressure probes.
 - 6.3.9 *Positioning of the Probe*:
- 6.3.9.1 In general, place the instrument probe perpendicular to the specimen surface at the point of measurement. For some instruments of the attractive force type, 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.
- 6.3.9.2 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 instrument is to be used in horizontal or upside-down position, calibrate it for that position.

7. Nickel-Phosphorus Alloys

- 7.1 Autocatalytic deposited nickel-phosphorus alloys containing more than 8 % phosphorus are sufficiently nonmagnetic to be measured in accordance with Test Method B 499, if deposited on a magnetic substrate.
- 7.2 Nickel-phosphorus alloys with less than 8 % phosphorus or that have been appropriately heat treated are more magnetic, but since their magnetic properties depend on the phosphorus content and on the heat treatment, their measurement by a magnetic method is not recommended and is not covered by this test method.

8. Report

- 87.1 The report should include the following information:
- 87.1.1 A reference to this standard,
- 7.1.2 Type of instrument used,
- 87.1.23 Size and description of test specimen,
- 87.1.34 Whether special jigs were used,
- 87.1.45 Type of calibration standard and the method used,
- 87.1.56 Thickness of the coating as determined from the measurements,
- 87.1.67 Operator identification, and
- 87.1.78 Date.

98. Precision and Bias

- 98.1 The equipment, its calibration, and its operation shall be such that the coating thickness can be determined with an uncertainty of less than 10 % at 95 % confidence level.
- 98.2 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 μm.
- 98.3 Instruments suitable for compliance with 6.1 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.
- 98.4 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 calibration standard used to calibrate the instrument.
 - 98.5 The precision is being determined by round-robin testing.

10.

9. Keywords

109.1 coating thickness; magnetic method; nickel coatings; thickness; thickness testing

(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 calibrated 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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