Designation: B 578 – 87 (Reapproved 1999)

Standard Test Method for
Microhardness of Electroplated Coatings[1]

1. Scope

1.1 This test method covers the determination of the hardness of metallic coatings upon various substrates. The measurements are made with the Knoop indenter under a test load of 0.245 N (25 gf) or 0.981 N (100 gf).

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:*
E 384 Test Method for Microhardness of Materials[2]

3. Special Requirements

3.1 In addition to the requirements listed herein, the test method shall be performed in accordance with Test Method E 384.

3.1.1 The measurement shall be made on the cross section with the long diagonal of the indentation parallel to the substrate using a Knoop indenter.

NOTE 1—When a nondestructive test is necessary, the measurement may be made normal to the surface provided that the whole of the indentation is clearly delineated in the field of the microscope at the magnification specified in Test Method E 384, and the coating thickness is at least 0.7 that of the long diagonal. The outlines of the indentation must be sharp and undistorted by any mechanical effects resulting from the thinness of the coating such as "butterfly" fractures, etc. Values obtained from measurements made normal to the electroplated surface may not be equivalent to those obtained on microsections and are not, therefore, reportable according to Section 6.

3.1.2 The thickness of the coating shall be at least 38 μm for soft coatings such as gold, copper, and silver, and 25 μm for hard coatings such as nickel, cobalt, iron, and hard gold. This is approximately equivalent to 0.6 times the length of the long Knoop diagonal.

3.1.3 Make the indentation near the center of the cross section of the coating.

3.2 No single test load is applicable to all coatings. Use a load of 0.245 N for soft coatings (from 50 to 300 HK) and a load of 0.981 N for hard coatings (greater than 300 HK).

3.2.1 A load of less than 0.245 N is not recommended. However, when the length of the diagonal is great enough at a load of 0.245 N, which may occur for very soft coatings such as tin and indium, a lighter load or a lower magnification shall be specified provided the requirements of 3.3 are met. When testing extremely hard coatings, a load greater than 0.981 N may be used provided this is specified in the purchase order. The Knoop hardness number decreases appreciably with loads from 0.245 N to 0.981 N especially for the higher hardness numbers.

3.3 Measure the length of the long diagonal to within 0.25 μm or 0.4 %, whichever is larger, at the magnification specified in Test Method E 384.

3.4 When readings are taken in a direction parallel to the substrate, space them from each other by at least the length of the long diagonal. When two readings are taken in the direction of the short axis, separate them by at least the length of the diagonal perpendicular to the substrate (short diagonal). In such cases, the distance from the substrate and from the outer surface of the coating to an edge of the indentation shall be at least the width of the short diagonal.

3.5 If the electroplated coating is cracked, or if cracks or "butterfly" markings (Test Method E 384, 7.2) appear when the indentation load is applied or removed, the hardness measurement is invalid.

4. Preparation of Test Specimen

4.1 Overplate the specimen to a thickness of at least 12 μm with a metal of a contrasting color whose hardness approximates that of the coating.

4.2 Cut out a section of the specimen approximately perpendicular to the electroplated coating.

4.3 Mount and polish the section as described in Test Method E 384.

NOTE 2—Procedures 4.1 and 4.2 are interchangeable at the discretion of the operator. A light etch following polishing may remove significant work hardening.

4.4 When the electrodeposit is a metal which may alloy with the basis metal (for example, indium over copper or copper alloys), or if the deposit has a low transformation temperature (such as nickel-phosphorus alloys), a low-temperature mounting procedure shall be used.

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5. Procedure

5.1 Insert the mounted specimen into the testing machine so that the test surface is parallel to the long axis of the indenter, and measure the hardness in accordance with the manufacturer's instructions and in accordance with Test Method E 384, Section A.

6. Report

6.1 Test results shall be based on at least five measurements, and the report shall include the following statistics:

6.1.1 The range, that is, the difference, in hardness units, between the highest and lowest values.

6.1.2 The average (arithmetic mean):

$$\bar{x} = \Sigma x/n \tag{1}$$

6.1.3 The estimated standard deviation:

$$s = \sqrt{\Sigma(x - \bar{x})^2/(n - 1)} \text{ or } \sqrt{(n\Sigma x^2 - (\Sigma x)^2/n(n - 1)} \tag{2}$$

6.1.4 The coefficient of variation:

$$\nu = \frac{s}{\bar{x}} \tag{3}$$

where, in 6.1.2-6.1.4:

s = the standard deviation,
x = an individual value,
$\bar{x}$ = arithmetic mean of the set of individual values,
n = the number of measurements,
Σx^2 = the sum of the squares of all of the individual values,
$(\Sigma x)^2$ = the square of the total of the individual values, and
ν = the coefficient of variation.

6.2 Report the hardness as a Knoop hardness number (HK), and indicate the load by subscript notation expressed as the force, in newtons, divided by 0.00981 (a number equal to that which is obtained when the test force is expressed in grams-force). For example: 410 $HK_{1\,00}$, 30 HK_{50}, 200 $HK_{2\,5}$.

NOTE 3—The International Organization for Standardization Technical Committee 17 on Steel has agreed that the formula for calculation and the system of designation for hardness shall be such that the numerical value of the hardness number remains unaffected by the introduction of the SI unit of force, the newton, in place of the old unit, kgf (or gf).

7. Precision and Bias

7.1 *Precision*—An interlaboratory comparison program is now in progress which when completed will be the basis of a statement on precision.

7.2 *Bias*—There is no basis for defining the bias of this test method.

7.3 Reference hardness specimens of electroformed nickel along with certified hardness readings as found by the National Institute of Standards and Technology (NIST) are currently available.[3]

8. Test Coupons

8.1 If specifically permitted by the governing specification or the party requesting the test, test coupons may be used when the production parts have insufficient electroplating thickness for measurement. The values obtained may not reflect the "true" hardness of the thin coatings on production parts. However, the values may be useful when they correlate with other coating properties such as wear resistance. The test may serve as a useful tool for electroplating bath control, particularly in the case of coatings such as gold, for which the hardness will be sensitive to the composition of the bath and other electroplating variables. The electroplating conditions for test coupons such as current density, temperature, agitation, and solution composition, shall be kept as close as possible to those employed on production parts in the plating process under test.

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[3] Available from the Office of Reference Materials, NIST, Gaithersburg, MD 20899.