Designation: E384 – 22

Standard Test Method for Microindentation Hardness of Materials[1]

This standard is issued under the fixed designation E384; 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 U.S. Department of Defense.

1. Scope*

1.1 This test method covers determination of the microindentation hardness of materials.

1.2 This test method covers microindentation tests made with Knoop and Vickers indenters under test forces in the range from 9.8×10^{-3} to 9.8 N (1 to 1000 gf).

1.3 This test method includes an analysis of the possible sources of errors that can occur during microindentation testing and how these factors affect the precision, bias, repeatability, and reproducibility of test results.

1.4 Information pertaining to the requirements for direct verification and calibration of the testing machine and the requirements for the manufacture and calibration of Vickers and Knoop reference hardness test blocks are in Test Method E92.

NOTE 1—While Committee E04 is primarily concerned with metals, the test procedures described are applicable to other materials.

1.5 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1326 Test Method for Knoop Indentation Hardness of Advanced Ceramics

C1327 Test Method for Vickers Indentation Hardness of Advanced Ceramics

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

E92 Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials

E140 Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, Scleroscope Hardness, and Leeb Hardness

E175 Terminology of Microscopy (Withdrawn 2019)[3]

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E766 Practice for Calibrating the Magnification of a Scanning Electron Microscope

E1268 Practice for Assessing the Degree of Banding or Orientation of Microstructures

E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques

E2587 Practice for Use of Control Charts in Statistical Process Control

2.2 *ISO Standard:*[4]

ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories

[1] This test method is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.05 on Microindentation Hardness Testing. With this revision the test method was expanded to include the requirements previously defined in E28.92, Standard Test Method for Vickers Hardness Testing of Metallic Material that was under the jurisdiction of E28.06

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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] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.org.

***A Summary of Changes section appears at the end of this standard**

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calibrating, v*—determining the values of the significant parameters by comparison with values indicated by a reference instrument or by a set of reference standards.

3.2.2 *Knoop hardness number, HK, n*—an expression of hardness obtained by dividing the force applied to the Knoop indenter by the projected area of the permanent impression made by the indenter.

3.2.3 *Knoop indenter, n*—a rhombic-based pyramidal-shaped diamond indenter with edge angles of ∠ A = 172° 30' and ∠ B = 130° 0' (see Fig. 1).

3.2.4 *microindentation hardness test, n*—a hardness test using a calibrated machine to force a diamond indenter of specific geometry into the surface of the material being evaluated, in which the test forces range from 1 to 1000 gf (9.8×10^{-3} to 9.8 N), and the indentation diagonal, or diagonals, are measured with a light microscope after load removal; for any microindentation hardness test, it is assumed that the indentation does not undergo elastic recovery after force removal.

NOTE 2—Use of the term microhardness should be avoided because it implies that the hardness, rather than the force or the indentation size, is very low.

3.2.5 *verifying, v*—checking or testing the instrument to assure conformance with the specification.

3.2.6 *Vickers hardness number, HV, n*—an expression of hardness obtained by dividing the force applied to a Vickers indenter by the surface area of the permanent impression made by the indenter.

3.2.7 *Vickers indenter, n*—a square-based pyramidal-shaped diamond indenter with face angles of 136° (see Fig. 2).

3.3 *Formulae*—The formulae presented in 3.3.1 – 3.3.4 for calculating microindentation hardness are based upon an ideal tester and conditions. The measured value of the microindentation hardness of a material is subjected to several sources of errors. Based on Eq 1-9, variations in the applied force, geometrical variations between diamond indenters, and human errors in measuring indentation lengths will affect the precision of the calculated material hardness. The magnitude of the error that variations of each of these parameters have on the calculated value of a microindentation measurement is discussed in Section 10.

3.3.1 For Knoop hardness tests, in practice, test loads are in grams-force and indentation diagonals are in micrometers. The Knoop hardness number is calculated using the following:

$$HK = 1.000 \times 10^3 \times (P/A_p) = 1.000 \times 10^3 \times P/(c_p \times d^2) \quad (1)$$

or

$$HK = 14229 \times P/d^2 \quad (2)$$

$$c_p = \frac{\tan\frac{\angle B}{2}}{2\tan\frac{\angle A}{2}} \quad (3)$$

where:

P = force, gf,
d = length of long diagonal, μm,
A_p = projected area of indentation, μm ^{2}
$\angle A$ = included longitudinal edge angle, 172° 30’
$\angle B$ = included transverse edge angle, 130° 0’ (see Fig. 1 and,
c_p = indenter constant relating projected area of the indentation to the square of the length of the long diagonal, ideally 0.07028.

3.3.2 The Knoop hardness, kgf/mm^2 is determined as follows:

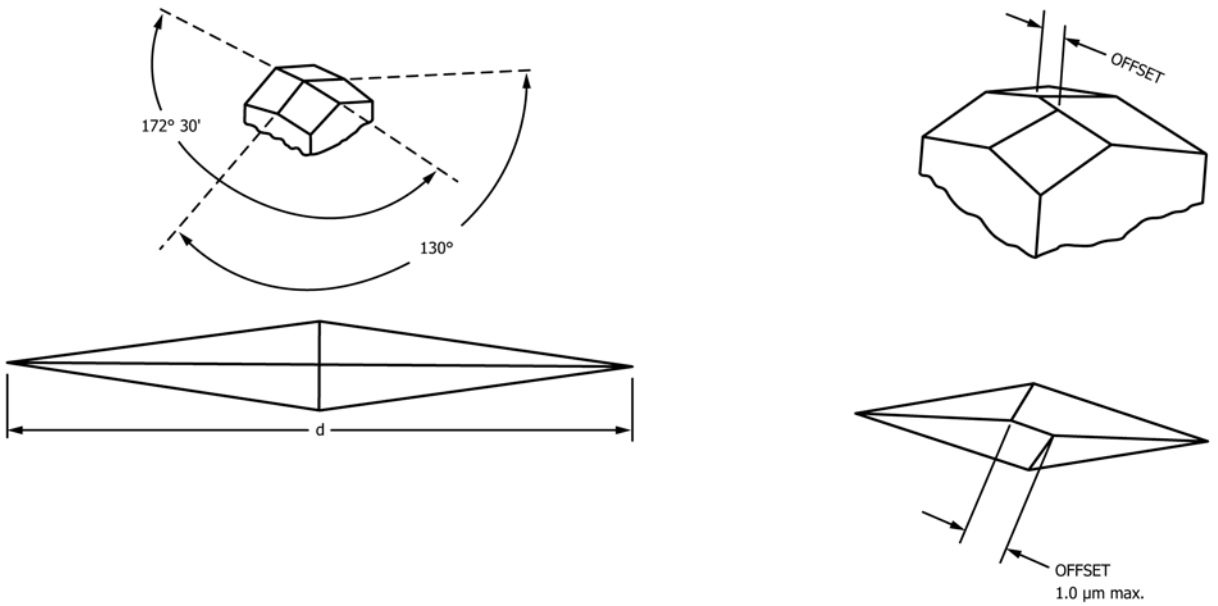

FIG. 1 Knoop Indenter

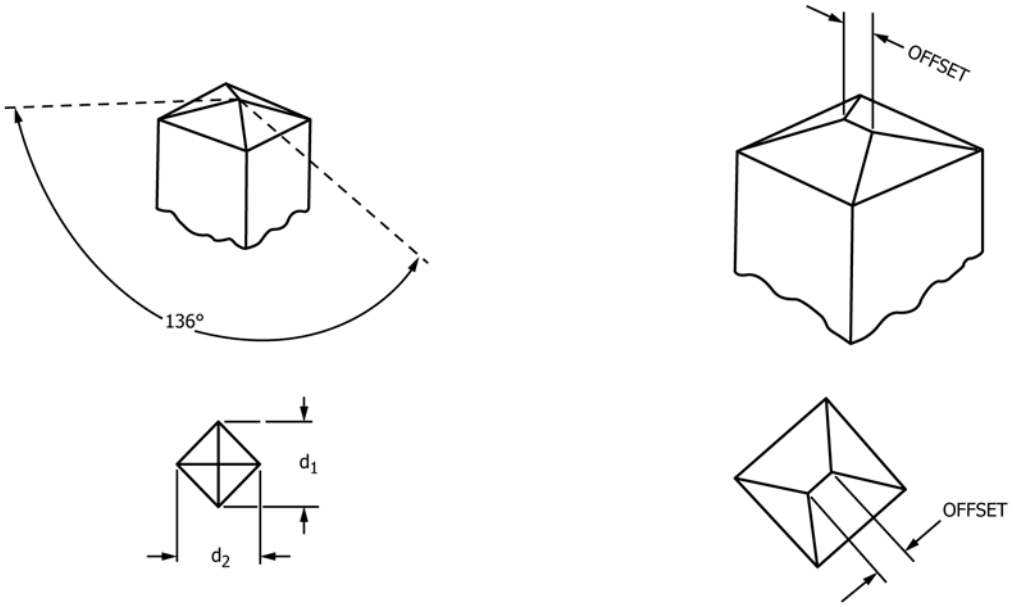

FIG. 2 Vickers Indenter

$$HK = 14.229 \times P_1/{d_1}^2 \quad (4)$$

where:

P_1 = force, kgf, and
d_1 = length of long diagonal, mm.

3.3.3 The Knoop hardness reported with units of GPa is determined as follows:

$$HK = 0.014229 \times P_2/{d_2}^2 \quad (5)$$

where:

P_2 = force, N, and
d_2 = length of the long diagonal of the indentation, mm.

3.3.4 For the Vickers hardness test, in practice, test loads are in grams-force and indentation diagonals are in micrometers. The Vickers hardness number is calculated as follows:

$$HV = 1.000 \times 10^3 \times P/A_s = 2.000 \times 10^3 \times P sin(\alpha/2)/d^2 \quad (6)$$

or

$$HV = 1854.4 \times P/d^2 \quad (7)$$

where:

P = force, gf,
A_s = surface area of the indentation, μm^2,
d = mean diagonal length of the indentation, μm, and
α = face angle of the indenter, 136° 0’ (see Fig. 2).

3.3.5 The Vickers hardness, kgf/mm^2 is determined as follows:

$$HV = 1.8544 \times P_1/{d_1}^2 \quad (8)$$

where:

P_1 = force, kgf, and
d_1 = mean diagonal length of the indentations, mm.

3.3.6 The Vickers hardness reported with units of GPa is determined as follows:

$$HV = 0.0018544 \times P_2/{d_2}^2 \quad (9)$$

where:

P_2 = force, N, and
d_2 = mean diagonal length of the indentations, mm.

3.4 Equations for calculating % Error and Repeatability for periodic verification is determined as follows:

$$E = 100\left(\frac{\left|\bar{d} - d_{ref}\right|}{d_{ref}}\right) \quad (10)$$

where:

E = % error in performance of the periodic verification,
$\bar{d}$ = the measured mean diagonal length in μm, and
d_{ref} = the reported certified mean diagonal length, μm.

$$R = 100\left(\frac{d_{\max} - d_{\min}}{\bar{d}}\right) \quad (11)$$

where:

R = repeatability in performance of the periodic verification,
d_{max} = the longest diagonal length measurement on the standardized test block, μm,
d_{min} = the shortest diagonal length measurement on the standardized test block, μm, and
$\bar{d}$ = the measured mean diagonal length in μm.

4. Summary of Test Method

4.1 In this test method, a hardness number is determined based on the formation of a very small indentation by application of a relatively low force, in comparison to traditional bulk indentation hardness tests.

4.2 A Knoop or Vickers indenter, made from diamond of specific geometry, is pressed into the test specimen surface under an applied force in the range of 1 to 1000 gf using a test machine specifically designed for such work.