Designation: C1326 − 13 (Reapproved 2023)

Standard Test Method for Knoop Indentation Hardness of Advanced Ceramics[1]

This standard is issued under the fixed designation C1326; 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 covers the determination of the Knoop indentation hardness of advanced ceramics. In this test, a pointed, rhombic-based, pyramidal diamond indenter of prescribed shape is pressed into the surface of a ceramic with a predetermined force to produce a relatively small, permanent indentation. The surface projection of the long diagonal of the permanent indentation is measured using a light microscope. The length of the long diagonal and the applied force are used to calculate the Knoop hardness which represents the material's resistance to penetration by the Knoop indenter.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *Units*—When Knoop and Vickers hardness tests were developed, the force levels were specified in units of grams-force (gf) and kilograms-force (kgf). This standard specifies the units of force and length in the International System of Units (SI); that is, force in newtons (N) and length in mm or µm. However, because of the historical precedent and continued common usage, force values in gf and kgf units are occasionally provided for information. This test method specifies that Knoop hardness be reported either in units of GPa or as a dimensionless Knoop hardness number.

1.4 *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.*

1.5 *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]

C730 Test Method for Knoop Indentation Hardness of Glass
C849 Test Method for Knoop Indentation Hardness of Ceramic Whitewares
E4 Practices for Force Calibration and Verification of Testing Machines
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E384 Test Method for Microindentation Hardness of Materials
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI) (The Modern Metric System)

2.2 *European Standard:*[3]

CEN ENV 843-4 Advanced Technical Ceramics, Monolithic Ceramics, Mechanical Properties at Room Temperature, Part 4: Vickers, Knoop, and Rockwell Superficial Hardness Tests

2.3 *ISO Standard:*[4]

ISO 9385 Glass and Glass Ceramics—Knoop Hardness Test

3. Terminology

3.1 *Definitions:*

3.1.1 *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.

3.1.2 *Knoop indenter, n*—a rhombic-based pyramidal-shaped diamond indenter with edge angles of 172° 30' and 130° 00'.

[1] This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.01 on Mechanical Properties and Performance.

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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 European Committee for Standardization (CEN), 36 rue de Stassart, B-1050, Brussels, Belgium, http://www.cenorm.be.

[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.ch.

4. Summary of Test Method

4.1 This test method describes an indentation hardness test using a calibrated machine to force a pointed, rhombic-based, pyramidal diamond indenter having specified face angles, under a predetermined force, into the surface of the material under test and measures the surface projection of the long diagonal of the resulting impression after removal of the load.

NOTE 1—A general description of the Knoop indentation hardness test is given in Test Method E384. The present test method differs from this description only in areas required by the special nature of advanced ceramics.

NOTE 2—This test method is similar to Test Methods C730 and C849, but differs primarily in the choice of force and the rate of force application. In addition, the length correction factor for the resolution limits of optical microscopes is not utilized.

5. Significance and Use

5.1 For advanced ceramics, Knoop indenters are used to create indentations. The surface projection of the long diagonal is measured with optical microscopes.

5.2 The Knoop indentation hardness is one of many properties that is used to characterize advanced ceramics. Attempts have been made to relate Knoop indentation hardness to other hardness scales, but no generally accepted methods are available. Such conversions are limited in scope and should be used with caution, except for special cases where a reliable basis for the conversion has been obtained by comparison tests.

5.3 For advanced ceramics, the Knoop indentation is often preferred to the Vickers indentation since the Knoop long diagonal length is 2.8 times longer than the Vickers diagonal for the same force, and cracking is much less of a problem (**1**).[5] On the other hand, the long slender tip of the Knoop indentation is more difficult to precisely discern, especially in materials with low contrast. The indentation forces chosen in this test method are designed to produce indentations as large as may be possible with conventional microhardness equipment, yet not so large as to cause cracking.

5.4 The Knoop indentation is shallower than Vickers indentations made at the same force. Knoop indents may be useful in evaluating coating hardnesses.

5.5 Knoop hardness is calculated from the ratio of the applied force divided by the projected indentation area on the specimen surface. It is assumed that the elastic springback of the narrow diagonal is negligible. (Vickers indenters are also used to measure hardness, but Vickers hardness is calculated from the ratio of applied force to the area of contact of the four faces of the undeformed indenter.)

5.6 A full hardness characterization includes measurements over a broad range of indentation forces. Knoop hardness of ceramics usually decreases with increasing indentation size or indentation force such as that shown in Fig. 1.[6] The trend is known as the indentation size effect (ISE). Hardness approaches a plateau constant hardness at sufficiently large indentation size or forces (loads). The test forces that are needed to achieve a constant hardness vary with the ceramic. The test force specified in this standard is intended to be sufficiently large that hardness is either close to or on the plateau, but not so large as to introduce excessive cracking. A comprehensive characterization of the ISE is recommended but is beyond the scope of this test method which measures hardness at a single, designated force.

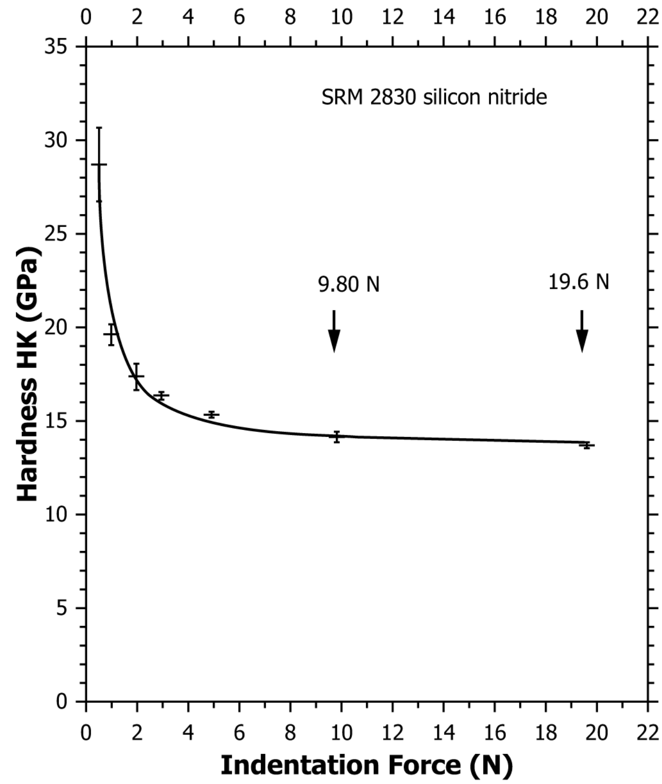

FIG. 1 A Typical Indentation Size Effect (ISE) Curve for a Ceramic (The data shown are for NIST SRM 2830 silicon nitride)

6. Interferences

6.1 Cracking from the indentation tips can interfere with interpretation of the exact tip location. The forces chosen for this test method are sufficiently low that tip cracking, if it occurs, will cause tiny, rather tight cracks at the indentation tips in advanced ceramics. Such cracks will have a negligible interference on measurements of the long diagonal length (**2**) (unlike Vickers indentations in ceramics).

6.2 Cracking or spalling from the sides of the Knoop impression may also occur, possibly in a time-dependent manner (minutes or hours) after the impression is made. Small amounts of such lateral cracking have little or no influence upon measured hardness, provided that the tip impressions are still readable and the tips are not dislodged (**2**).

6.3 Porosity (either on or just below the surface) may interfere with measuring Knoop hardness, especially if the indentation falls directly onto a large pore or if the indentation tip falls in a pore.

[5] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[6] Standard Reference Materials Program (NIST) 100 Bureau Drive, Stop 2300 Gaithersburg, MD 20899-2300.

6.4 At higher magnifications in the optical microscope, it may be difficult to obtain a sharp contrast between the indentation tip and the polished surface of some advanced ceramics. This may be overcome by careful adjustment of the lighting as discussed in Test Method E384 and Refs (**2, 3**).

7. Apparatus

7.1 *Testing Machines:*

7.1.1 There are three general types of machines available for making this test. One type is a self-contained unit built for this purpose that uses deadweights (masses) on a pan or lever beam to carefully apply force to the test piece. There is no load cell to record the force during the test sequence. The machine has a built-in compound optical microscope for measuring the indentation sizes. The second type is an accessory to existing compound optical microscopes. Usually, this second type is fitted on an inverted-stage microscope. The third, more modern type, is a self-contained unit built for this purpose which has a built-in load cell that controls a ram or crosshead that moves the indenter into contact with the test piece. The peak force and rate of force application can be controlled by a closed-loop feedback circuit. The machine has a built-in compound optical microscope for measuring the indentation sizes. Descriptions of the various machines are available (**4-6**).

7.1.2 Design of the machine should be such that the loading rate, dwell time, and applied load can be set within the limits set forth in 10.5. It is an advantage to eliminate the human element whenever possible by appropriate machine design. The machine should be designed so that vibrations induced at the beginning of a test will be damped out by the time the indenter touches the sample.

7.1.3 The calibration of the balance beam or force application system should be checked monthly or as needed. Indentations in standard reference materials may also be used to check calibration when needed.

7.2 *Indenter:*

7.2.1 The indenter shall meet the specifications for Knoop indenters. See Test Method E384.

7.2.2 Fig. 2 shows the indenter and its maximum usable dimensions. The diagonals have an approximate ratio of 7:1, and the depth of the indentation is approximately 1⁄30 the length of the long diagonal. A perfect Knoop indenter has the following angles:

7.2.2.1 Included longitudinal angle 172° 30 min 00 s.

7.2.2.2 Included transverse angle 130° 00 min 00 s.

7.2.3 The constant C_p (defined in 12.2) for a perfect indenter is 0.07028. The specifications require a variation of not more than 1 % from this value.

7.2.4 The offset at the indenter tip shall not exceed 1.0 µm. See Test Method E384.

7.2.5 The four faces of the indenter shall meet at sharp edges.

7.2.6 The diamond should be examined periodically, and if it is loose in the mounting material, chipped, or cracked, it shall be replaced.

NOTE 3—This requirement is from Test Method E384 and is especially pertinent to diamond indenters that are used to measure hardness of ceramics. In addition, these indenters sometimes are used to precrack advanced ceramic specimens at loads higher than customarily used for hardness testing. Such usage can lead to indenter damage. The diamond indenter can be examined with a scanning electron microscope, or indents can be made into soft copper to help determine if a chip or crack is present. Indenters may also be inspected with an optical microscope with at least 500× power, but care should be taken to avoid damaging the microscope lens.

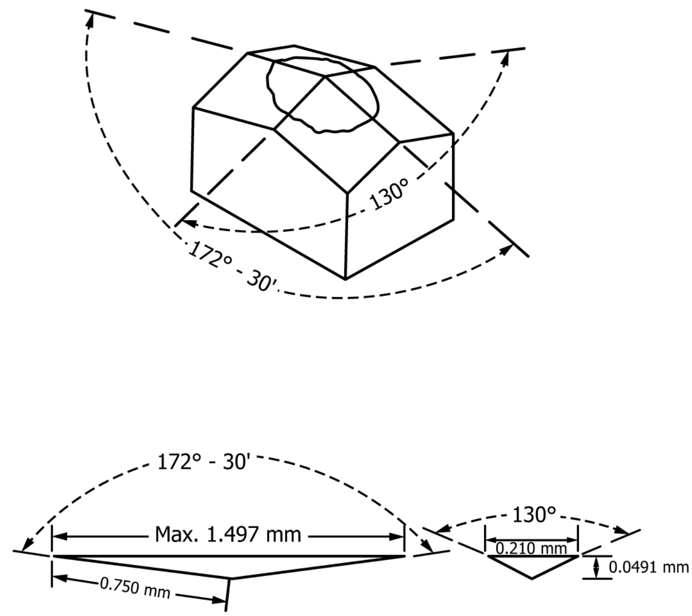

FIG. 2 Knoop Indenter Showing Maximum Usable Dimensions

7.3 *Measuring Microscope:*

7.3.1 The measurement system shall be constructed so that the length of the diagonals can be determined with errors not exceeding ±0.0005 mm.

NOTE 4—Stage micrometers with uncertainties less than this shall be used to establish calibration constants for the microscope. See Test Method E384. Ordinary stage micrometers which are used for determining the approximate magnification of photographs may be ruled too coarse or may not have the required accuracy and precision.

7.3.2 The numerical aperture (NA) of the objective lens shall be between 0.60 and 0.90.

NOTE 5—The apparent length of a Knoop indentation will increase as the resolving power and NA of a lens increases. The range of NA specified by this test method corresponds to 40 to 100× objective lenses. The higher-power lenses may have higher resolution, but the contrast between the indentation tips and the polished surface may be less.

7.3.3 A filter may be used to provide monochromatic illumination. Green filters have proved to be useful.

8. Test Specimens

8.1 The Knoop indentation hardness test is adaptable to a wide variety of advanced ceramic specimens. In general, the accuracy of the test will depend on the smoothness of the surface and, whenever possible, ground and polished specimens should be used. The back of the specimen shall be fixed so that the specimen cannot rock or shift during the test.

8.1.1 *Thickness*—As long as the specimen is over ten times as thick as the indentation depth, the test will not be affected. In general, if specimens are at least 0.50 mm thick, the hardness will not be affected by variations in the thickness.