Standard Practice for
Instrumented Indentation Testing[1]

1. Scope*

1.1 This practice defines the basic steps of Instrumented Indentation Testing (IIT) and establishes the requirements, accuracies, and capabilities needed by an instrument to successfully perform the test and produce the data that can be used for the determination of indentation hardness and other material characteristics. IIT is a mechanical test that measures the response of a material to the imposed stress and strain of a shaped indenter by forcing the indenter into a material and monitoring the force on, and displacement of, the indenter as a function of time during the full loading-unloading test cycle.

1.2 The operational features of an IIT instrument, as well as requirements for Instrument Verification (Annex A1), Standardized Reference Blocks (Annex A2) and Indenter Requirements (Annex A3) are defined. This practice is not intended to be a complete purchase specification for an IIT instrument.

1.3 With the exception of the non-mandatory Appendix X4, this practice does not define the analysis necessary to determine material properties. That analysis is left for other test methods. Appendix X4 includes some basic analysis techniques to allow for the indirect performance verification of an IIT instrument by using test blocks.

1.4 Zero point determination, instrument compliance determination and the indirect determination of an indenter's area function are important parts of the IIT process. The practice defines the requirements for these items and includes non-mandatory appendixes to help the user define them.

1.5 The use of deliberate lateral displacements is not included in this practice (that is, scratch testing).

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]
E3 Guide for Preparation of Metallographic Specimens
E74 Practice of Calibration of Force-Measuring Instruments for Verifying the Force Indication of Testing Machines
E92 Test Method for Vickers Hardness of Metallic Materials (Withdrawn 2010)[3]
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E384 Test Method for Knoop and Vickers Hardness of Materials
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1875 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio by Sonic Resonance
E1876 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio by Impulse Excitation of Vibration

2.2 *American Bearing Manufacturers Association Standard:*
ABMA/ISO 3290-1 Rolling Bearings- Balls-Part 1: Steel Metal Balls[4]

2.3 *ISO Standards:*
ISO 14577-1, -2, -3, -4 Metallic Materials—Instrumented Indentation Tests for Hardness and Material Properties[5]
ISO 376 Metallic Materials—Calibration of Force-Proving Instruments for the Verification of Uniaxial Testing Machines[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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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 American Bearing Manufacturers Association (ABMA), 2025 M Street, NW Suite 800 Washington, DC 20036, http://www.americanbearings.org.

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

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

3.1.1 *contact stiffness, n*—the instantaneous elastic response of the material over the area of contact with the indenter.

3.1.1.1 *Discussion*—Contact stiffness can be determined from the slope of line 3 in Fig. 1.

3.1.2 *force displacement curve, n*—a common plot of the force applied to an indenter and the resultant depth of penetration.

3.1.2.1 *Discussion*—This plot is generated from data collected during the entire loading and unloading cycle. (See Fig. 1.)

3.1.3 *indentation radius [a], n*—the in-plane radius, at the surface of the test piece, of the circular impression of an indent created by a spherical indenter.

3.1.3.1 *Discussion*—For non-circular impressions, the indentation radius is the radius of the smallest circle capable of enclosing the indentation. The indentation radius is normally used as a guide for spacing of indentations.

3.1.4 *indenter area function [Λ], n*—mathematical function that relates the projected (cross-section) area of the indenter tip to the distance from the apex of the tip as measured along the central axis.

3.1.5 *instrument compliance, n*—the flex or reaction of the load frame, actuator, stage, indenter, anvil, etc., that is the result of the application of a test force to the sample.

3.1.6 *instrumented indentation test (IIT), n*—an indentation test where the force applied to an indenter and the resultant displacement of the indenter into the sample are recorded during the loading and unloading process for post test analysis.

3.1.7 *nominal area function, n*—area function determined from measurement of the gross indenter geometry.

3.1.8 *refined area function, n*—area function determined indirectly by a technique such as the one described in Appendix X3.

3.1.9 *test cycle, n*—a series of operations at a single location on the test sample specified in terms of either applied test force or displacement as a function of time.
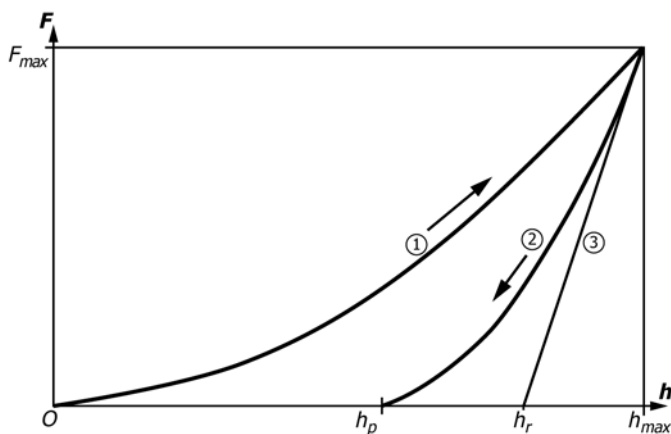
3.1.9.1 *Discussion*—The test cycle may include any of the following operations: approach of the indenter towards the test sample, singular or multiple loading, dwell, and unloading cycles.

3.1.10 *test data, n*—for this practice it will consist, at the minimum, of a set of related force/displacement/time data points.

3.1.11 *zero point, n*—the force-displacement-time reference point when the indenter first contacts the sample and the force is zero.

3.1.11.1 *Discussion*—A course zero point is an approximate value used as part of an analysis to determine a refined value.

3.2 *Indentation Symbols and Designations* (see Fig. 2 and Table 1):

4. Summary of Practice

4.1 This practice defines the details of the IIT test and the requirements and capabilities for instruments that perform IIT tests. The necessary components are defined along with the required accuracies required to obtain useful results. Verification methods are defined to insure that the instruments are performing properly. It is intended that ASTM (or other) Test Methods will refer to this practice when defining different calculations or algorithms that determine one or more material characteristics that are of interest to the user.

5. Significance and Use

5.1 IIT Instruments are used to quantitatively measure various mechanical properties of thin coatings and other volumes of material when other traditional methods of determining material properties cannot be used due to the size or condition of the sample. This practice will establish the basic requirements for those instruments. It is intended that IIT based test methods will be able to refer to this practice for the basic requirements for force and displacement accuracy, reproducibility, verification, reporting, etc., that are necessary for obtaining meaningful test results.
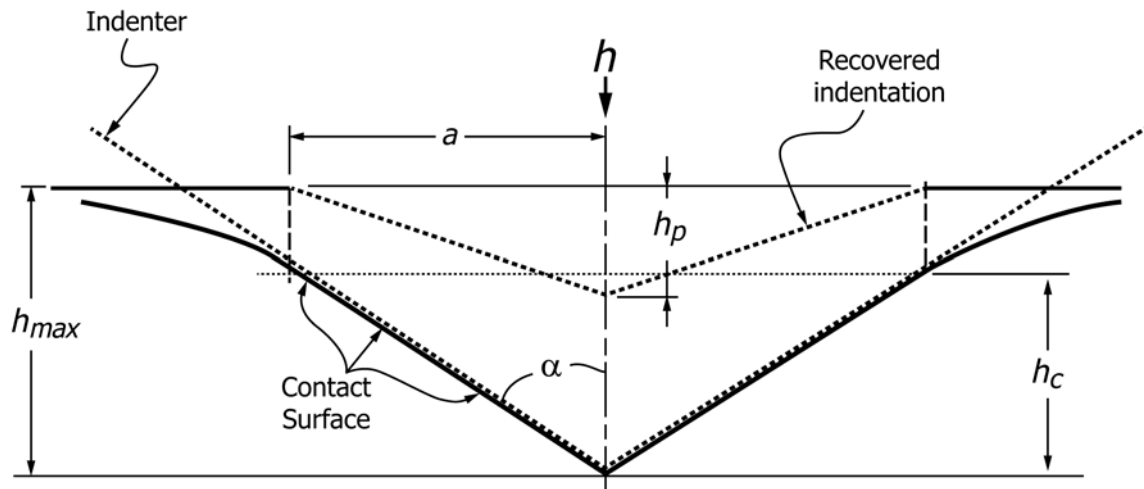
5.2 IIT is not restricted to specific test forces, displacement ranges, or indenter types. This practice covers the requirements for a wide range of nano, micro, and macro (see ISO 14577-1) indentation testing applications. The various IIT instruments are required to adhere to the requirements of the practice within their specific design ranges.

6. Apparatus

6.1 *General*—The force, displacement and time are simultaneously recorded during the full sequence of the test. An analysis of the recorded data must be done to yield relevant information about the sample. When available, relevant ASTM test methods for the analysis should be followed for comparative results.

NOTE 1—The user is encouraged to refer to the manufacturer's instruction manual to understand the exact details of the tests and analysis performed.

6.2 *Testing Instrument*—The instrument shall be able to be verified according to the requirements defined in Annex A1 and have the following features.

1. Increasing test force
2. Removal of the test force
3. Tangent to curve 2 at F_{max}

FIG. 1 IIT Procedure Shown Schematically

NOTE 1—The symbols shown are the same for pointed and spherical indenters.
FIG. 2 Schematic Cross-Section of an IIT Indentation

TABLE 1 Symbols and Designations

Symbol	Designation	Unit
α	Angle, specific to shape of pyramidal indenter (see Annex A3)	°
a	Radius of indentation (see 3.1.3)	µm
R	Radius of spherical indenter (see Annex A3)	µm
F	Test force applied to sample	N
F_{max}	Maximum value of F	N
h	Indenter displacement into the sample	µm
h_{max}	Maximum value of h	µm
h_c	Depth over which the indenter and specimen are in contact during the force application	µm
h_p	Permanent recovered indentation depth after removal of test force	µm
A_s	Surface area of indenter in contact with material	µm²
A_p	Projected (cross section) area of indenter at depth h_c	µm²
h_r	Point of intersection of line 3 with the h axis (see Fig. 1)	µm
S	Contact stiffness	N/µm
t	Time relative to the zero point	s

6.2.1 *Test Forces/Displacements*—The instrument shall be able to apply operator selectable test forces or displacements within its usable range. The controlled parameters can vary either continuously or step by step. The application of the test force shall be smooth and free from any unintended vibrations or abnormalities that could adversely affect the results. The approach, loading, and data acquisition rates shall be controlled to the extent that is required to obtain meaningful estimates of force and displacement uncertainties at the zero point. The estimated uncertainty in the force at the zero point shall not exceed 1 % of the maximum test force (F_{max}) or 2 µN, whichever is greater. The estimated uncertainty in the displacement at the zero point should not exceed 1 % of the maximum indenter displacement (h_{max}) or 2 nm, whichever is greater. If the estimated uncertainty in the displacement at the zero point is larger than both criteria, its value and the influence of its value on reported mechanical properties shall be noted in the test report. See Appendix X1 for information on how to determine the zero point.

6.2.2 *Sample Positioning*—The positioning of the sample being tested relative to the centerline of the test force is critical to obtaining good results. The testing instrument shall be designed to allow the centerline of the test force to be normal to the sample surface at the point of indentation.

6.2.3 *Indenters*—Indenters normally consists of a contact tip and a suitable holder. The tip should have a hardness and modulus that significantly exceeds the materials being tested. The holder shall be manufactured to support the contact point without any unpredictable deflections that could affect the test results. The holder shall allow proper mounting in the actuator and position the contact point correctly for the application of the test force. The contact tip and holder could be a one or multi-piece design. A variety of indenter shapes, such as pyramids, cones, and spheres, can be used for IIT Testing. Annex A3 defines the requirements for the most commonly used indenters. Whenever they are used the requirements of Annex A3 shall be followed. Other indenter shapes can be used provided they are defined in a standardized Method or described in the test report.

NOTE 2—The nominal indenter geometry, as described in Annex A3, may be sufficiently accurate for a given analysis. In many cases, however, a refined area function that more accurately represents the shape of the indenter used may be necessary to provide the desired results (see A3.7).

6.2.4 *Imaging Device (Optional)*—In applications where it is desirable to accurately locate the indentation point on the sample or observe the indent, an imaging device such as an optical or atomic force microscope may prove helpful. The device should be mounted such that locations can be identified quickly and accurately.

6.3 *Data Storage and Analysis Capabilities*—The apparatus shall have the following capabilities:

6.3.1 *Force/Displacement/Time Measurement*—Acquire and store raw force, displacement and time data during each test.

6.3.2 *Data Correction*—When necessary, conversion of the raw data defined in 6.3.1 to corrected force (F), displacement (h), and time (t) data as defined in 3.2. The conversion shall

consider at least the following parameters: Zero point determination (see Appendix X1), instrument compliance (see Appendix X2) and thermal drift.

6.3.3 *Indenter Shape Function*—Utilize an appropriate indenter shape function if necessary (see Appendix X3).

6.3.4 *Test Result Generation:*

6.3.4.1 Perform the desired analysis on the raw or corrected data to obtain useful test results. When available, relevant ASTM or ISO 14577 test methods should be used.

6.3.4.2 Determine indentation modulus (E_{IT}) according to the Test Method defined in Appendix X4 or another method that produces similar results.

7. Test Piece

7.1 *Surface Finish*—The surface finish of the sample will directly affect the test results. The test should be performed on a flat specimen with a polished or otherwise suitably prepared surface. Any contamination will reduce the precision and accuracy of the test. The user should consider the indent size when determining the proper surface finish.

7.2 *Surface Preparation*—The preparation of the surface shall be done in a way that minimizes alteration of the characteristic of the material to be evaluated.

7.3 *Sample Thickness*—The thickness of the material to be analyzed may be a critical factor in the ability to obtain the desired results. The test piece thickness shall be large enough, or indentation depth small enough, such that the test result is not influenced by the test piece support. The test piece thickness should be at least 10 times the indentation depth or six times greater than the indentation radius; whichever is greater.

8. Procedure

8.1 *Prepare Environment*—The test should be carried out within the temperature range defined by the manufacturer. Prior to performing any tests the instrument and the test sample shall be stabilized to the temperature of the environment. Temperature change during each test should be less than 1.0°C. The test environment shall be clean and free from vibrations, electromagnetic interference, or other variations that could adversely affect the performance of the instrument. Testing done outside the specified limits is allowed; however, all deviations shall be specified on the test report.

8.2 *Mount Specimen*—The sample shall be rigidly supported and the test surface shall be positioned normal to the centerline of the test force.

NOTE 3—Sample fixtures may add to the compliance of the instrument. The user should consider the impact of this undesirable effect.

8.3 *Select Test Location*—The results of indentation tests will be adversely affected if the properties being measured vary within the volume of material being deformed. Extreme conditions would be caused by the presence of free surfaces such as edges, voids and other indentations. For many materials it is sufficient to locate the test at least six indent radii away

from such features; however, there are exceptions to this rule. The measurements of elastic properties, for example, are significantly more sensitive and require greater spacing than those for plastic properties. It is the responsibility of the operator to exercise caution so that such gradients do not affect the desired results.

8.4 *Define the Test Cycle*—The test cycle parameters shall be chosen with respect to the following considerations:

8.4.1 The forces generated by the dynamic motion of the indenter mass shall not adversely affect the accuracy of the results. This is particularly true at the point of contact when the intentionally applied forces are small.

8.4.2 The test cycle force and displacement values used in the test result analysis, except those used for zero point determination, shall be within the verified range of the instrument as reported in A1.7.2.4 and A1.7.2.5.

8.5 *Perform the Test Cycle*—The test cycle (see 3.1.9) is performed according to the specifications of the manufacturer or the test method. Force/displacement/time data shall be acquired during each test cycle.

8.6 *Correct the Data*—The acquired data shall be corrected according to 6.3.2. The details may be defined by the manufacturer or by a test method.

8.7 *Analyze Results*—The corrected data shall be analyzed to obtain the desired test results according to 6.3.4. The details may be defined by the manufacturer or by a test method.

9. Report

9.1 The report shall include sufficient information about the test cycle, indenter, sample and analysis method used to allow the final results to be reproduced.

9.2 The report shall include the following minimum information:

9.2.1 Date and time,

9.2.2 Reference to this practice,

9.2.3 Description of instrument—mfg., model, etc.,

9.2.4 Shape and material of the indenter used,

9.2.5 Temperature,

9.2.6 Test sample description,

9.2.7 Description of test cycle,

9.2.8 Method of zero point determination,

9.2.9 Reference to analytic method used, including values of any model dependent parameters,

9.2.10 Number of tests and results,

9.2.11 Details of any occurrence that may have affected the results, and

9.2.12 Define the units of the test results.

NOTE 4—It is also frequently desirable to describe the location of the indentation on the test piece as part of the report.

10. Keywords

10.1 force displacement curve; indentation hardness; indentation modulus; indenter shape function; instrument compliance; instrumented indentation; zero point

ANNEXES

(Mandatory Information)

A1. INSTRUMENT VERIFICATION

A1.1 Scope

A1.1.1 This annex specifies procedures for verification of testing machines that conform to the requirements defined in this practice. The annex describes a direct verification procedure for checking the main functions of the testing machine and an indirect verification procedure suitable for assessing the overall performance of the testing machine. The indirect procedure is used as part of the direct procedure and for the periodic routine checking of the machine in service. This annex does not cover verification procedures for specific indenter geometry; such procedures are presented in Annex A3 Indenter Requirements. The manufacturer's recommendations concerning instrument calibration and verification should be used as long as they do not conflict with the specifications of this annex.

A1.2 General Conditions

A1.2.1 Direct and indirect verification procedures shall be carried out at a temperature of 23 ± 5°C.

Note A1.1—For both verification and operation, thermal stability of the instrument is important. During any verification procedure, reasonable care should be taken to ensure that the temperature of the instrument and its immediate environment are kept at a constant temperature, preferably to within a 0.5°C range over the course of the verification procedure or test.

A1.3 Direct Verification

A1.3.1 Direct verification requires assessment of: *(1)* Force, *(2)* Displacement, and *(3)* Timing. If available, the devices used for force and displacement verification shall be traceable to National Standards.

A1.3.2 *Force Verification*—Each force range of the instrument shall be verified as described below.

A1.3.2.1 At least ten verification forces shall be chosen that evenly span the defined force range. The measurement of each verification force shall be repeated three times. Every measurement shall be within 1 % or 2 μN of its nominal value, whichever is greater. When the 2 μN tolerance is used the maximum test force (F_{max}) shall be accurate to within 5 % of the stated value. The verified force range of an instrument shall be defined as the range of forces from the minimum verified force to the maximum verified force.

A1.3.2.2 The device used to verify forces shall be accurate to within 0.25 % or 1 μN of each verification force, whichever is greater. Examples of techniques for force verification include:

(1) Measuring by means of an elastic proving device in accordance with Practice E74 (class A), or ISO 376 (class 1),

(2) Balancing against a force, applied by means of calibrated masses, and

(3) Measuring by means of an electronic balance.

A1.3.2.3 If the verification force is applied in the opposite direction from the force used during a test, the manufacturer shall provide documentation confirming that the verification results would be within the tolerance if the force were applied in the test direction.

A1.3.2.4 If the force calibration is assumed to be independent of indenter position, the manufacturer shall show that this is the case.

A1.3.3 *Displacement Verification*—Each displacement range of the instrument shall be verified as described below.

A1.3.3.1 At least ten verification lengths shall be chosen so as to span evenly the defined displacement range. The measurement of each verification length shall be repeated three times. Every measurement shall be within 1 % or 2 nm of its nominal value, whichever is greater. If the 2nm tolerance is used the maximum indenter displacement (h_{max}) shall be accurate to within 5 % of the stated value. The verified displacement range of an instrument shall be defined as the range of lengths from the minimum verified length to the maximum verified length.

A1.3.3.2 The device used for displacement verification shall be accurate to within 0.25 % or 1 nm of each verified length, whichever is greater. Methods of producing lengths for verification include:

(1) Laser interferometers,

(2) Film thickness standards, and

(3) Independent actuator or transducer.

A1.3.4 *Timing Verification*—The time required for a test segment at least ten seconds in duration shall be verified by an independent timing device. The difference between the time reported by the test equipment and that measured by an independent timing device must be less than 1.0 seconds. A hand-operated, non-traceable, stopwatch is sufficient for this verification.

A1.4 Indirect Verification

A1.4.1 An independent indirect verification shall be performed for each force range of the instrument. Indirect verification is intended to monitor the total performance of the instrument including force and displacement calibrations, machine compliance, indenter shape function, method of zero-point determination, and the analysis procedures. Therefore, these parameters shall remain fixed during indirect verification.

A1.4.2 *Procedure*—Indirect verification requires determining the indentation modulus, E_{IT}, of two materials of known Young's modulus. The two material's Young's modulus shall differ by at least a factor of two. Test blocks that comply with Annex A2 of this practice, should be used.

Note A1.2—The Test Method defined in Appendix X4 is recommended for this procedure.

A1.4.2.1 On each material, five tests shall be performed within each of the following force ranges:

(1) A maximum force within the lower 25 % of the verified force range, and

(2) A maximum force within the upper 25 % of the verified force range.

A1.4.2.2 The instrument is considered verified if ninety percent of the values of indentation modulus reported by the test equipment match the nominal Young's modulus, or the dynamic Young's modulus, value assigned to the material to within ±5 %.

A1.4.3 *Failure of the Indirect Verification*—When the results of the indirect verification are unsatisfactory, the manufacturers' guidelines for troubleshooting should be followed, and the indirect verification repeated. If the results are still not satisfactory, the instrument fails indirect verification.

A1.5 Routine Checking

A1.5.1 Routine checking shall be used according to the schedule defined in A1.6.3 to monitor the performance of the instrument.

A1.5.2 *Procedure*—Routine checking requires performing at least three tests on a single material of known indentation modulus. The test parameters, such as maximum force and displacement, should be similar to those that will be used until the next routine check. Eighty percent of the values of indentation modulus reported by the instrument shall match the expected value to within ±5 %.

NOTE A1.3—The test method used may require additional testing on test blocks or other reference materials unique to the method.

A1.5.3 *Failure of Routine Checking*—When the results of routine checking are unsatisfactory, the manufacturers' guidelines for troubleshooting should be followed, and the routine check repeated. If the results are still not satisfactory, the instrument fails routine checking.

A1.6 Verification Schedule

A1.6.1 *Direct Verification*—Direct verification shall be performed:

A1.6.1.1 When the instrument is first certified to comply with this standard,

A1.6.1.2 Following a major repair or overhaul of the instrument, including replacement of a component in the force or displacement system, except as described in A1.6.2 of this annex, and

A1.6.1.3 When the instrument fails indirect verification as defined in A1.4 of this annex.

NOTE A1.4—It is recommended that direct verification be performed upon installation of an instrument at a new location and at intervals not to exceed three years. Instruments intended for portable use cannot easily be directly verified at each location; therefore they should be verified at a known stable location.

A1.6.2 *Indirect Verification*—Indirect verification shall be performed:

A1.6.2.1 Following direct verification,

A1.6.2.2 After any relocation of the instrument, except for instruments designed specifically for portable use,

A1.6.2.3 At intervals not to exceed one year,

A1.6.2.4 When the machine fails routine checking as defined in A1.6.3 of this annex, and

A1.6.2.5 When the correction for machine compliance is changed.

NOTE A1.5—If the correction for machine compliance is known to change in a predictable way, that is, as a function of mounting type or sample position, the machine compliance correction may be changed according to a pre-established algorithm. An indirect verification is not required after such a change.

A1.6.2.6 Following the replacement of a component in either the force or displacement measurement system, provided that the manufacturer can show that such a replacement does not affect the force or displacement calibration of the complete machine.

NOTE A1.6—It is recommended that the indirect verification process be used to determine the indenter shape function for a specific indenter.

A1.6.3 *Routine Checking*—Routine checking shall be performed:

A1.6.3.1 Every day that the instrument is used,

A1.6.3.2 When an indenter is changed, and

A1.6.3.3 After changes in hardware that may affect the machine compliance.

A1.6.4 *Verification Flowchart*—A flowchart showing guidelines for verification of the various components is shown in Fig. A1.1.

A1.7 Reporting Results for Verifications and Routine Checking

A1.7.1 Results from all direct and indirect verifications, including out-of-tolerance data, shall be maintained in a log associated with the instrument.

A1.7.2 *Direct Verification Report*—Reporting for a direct verification shall include at least the following information:

A1.7.2.1 Reference to this standard,

A1.7.2.2 Identification data for the machine,

A1.7.2.3 Environmental temperature and humidity,

A1.7.2.4 Verified force range of the instrument, as well as verification forces used and measured values for those forces (see A1.3.2 of this annex),

A1.7.2.5 Verified displacement range of the instrument, as well as verification lengths used and measured values for those lengths (see A1.3.3 of this annex),

A1.7.2.6 Identification of devices used for force and displacement verification, including any relevant traceability information,

A1.7.2.7 Results of timing verification including nominal time of the test segment and measured time, and

A1.7.2.8 Name of the verification laboratory and date of verification.

A1.7.3 *Reporting for Indirect Verification*—Reporting for an indirect verification shall include all of the information required by standard reporting as described in Section 9. Test sample description shall include the nominal dynamic Young's modulus for the test materials.

A1.7.4 *Documentation of Routine Checking*—A formal report for routine checking is not required. However, it is recommended that a log of these results be maintained.
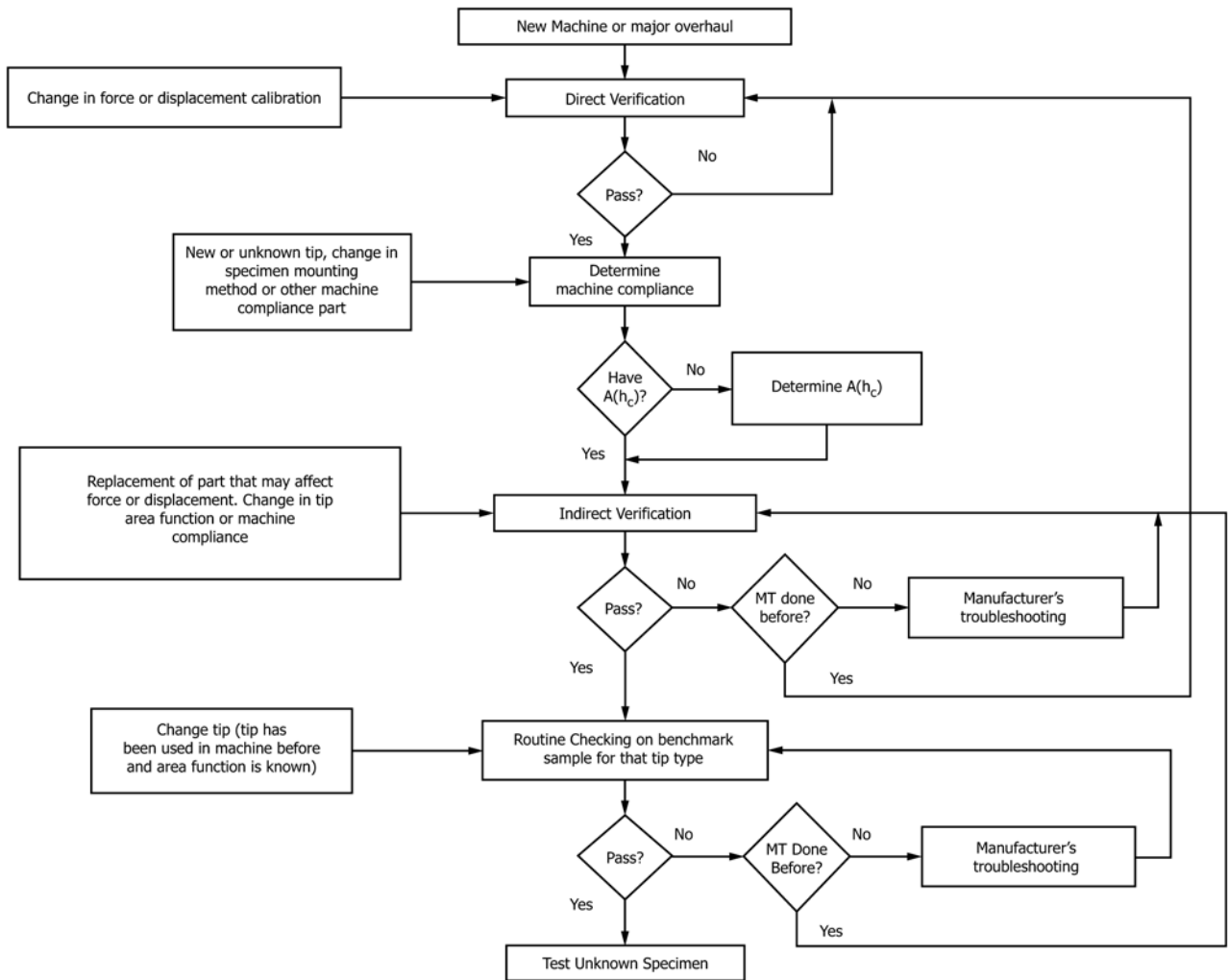
FIG. A1.1 Verification Flowchart

A2. STANDARD REFERENCE BLOCKS

A2.1 Scope

A2.1.1 This annex specifies requirements for the production and certification of standard reference blocks for use in the indirect verification of instrumented indentation instruments, as described in A1.6.2 of this practice.

A2.2 General Requirements

A2.2.1 Standard reference blocks shall be manufactured from materials with known values of dynamic Young's modulus, E, and Poisson's ratio, ν, each determined to an accuracy better than 1.0 %.

A2.2.2 Each test block shall be provided with certified values of dynamic Young's modulus and Poisson's ratio. Instrumented indentation tests shall be used to determine a usable range of depth or load for each block. Examples of material characteristics that might limit the range of appropriate indentation force include the cracking of a brittle material

above a certain force, or unacceptable scatter in indentation results below a certain indentation force as the result of surface roughness or small-scale nonhomogeneities.

NOTE A2.1—These minimum and maximum values for force or displacement will in general be indenter-specific. For example, maximum loads for blocks of brittle material may be much higher for large-radii spheres than for Berkovich tips.

A2.3 Material Selection

A2.3.1 Materials for standard reference blocks should have the following characteristics:

A2.3.1.1 A well-known, uniform composition,

A2.3.1.2 An amorphous or single crystal structure or known grain size distribution,

A2.3.1.3 Isotropic elastic properties,

A2.3.1.4 A chemically stable surface,

A2.3.1.5 A melting or glass transition temperature well above room temperature, and

A2.3.1.6 Little or no pile-up of material about the perimeter of the indentation site.

A2.4 Manufacture of Reference Blocks

A2.4.1 *Test Surface Orientation*—Reference blocks shall be manufactured in such a way that the test surface can be presented perpendicular to the indenter axis within 0.5 degrees. For specimens that are intended to be placed with their bottom surface (that surface opposite the test surface) on a specimen mounting plate, this requirement shall be met by achieving the necessary 0.5 degree parallelism between top and bottom surfaces. For specimens that are to be mounted by their sides (that is, a cylindrical reference block clamped in a V-shaped vise), the side surfaces shall be perpendicular to the test surface to within 0.5 degrees.

A2.4.2 *Test Surface Finish*—Test surface roughness can seriously degrade the accuracy and reproducibility of indentation test results. Therefore, reference blocks should be prepared in such a way that the test surface presented is as smooth as is possible for a given material. An acceptable value of average surface roughness, R_A, for many applications is $R_A \leq 10$ nm measured over a 10 μm trace. Blocks intended specifically for very-low-force verification will require lower roughness levels. For guidelines on the preparation on metallographic specimens, see for example Guide E3.

A2.4.3 *Reference Block Compliance*—In some cases, the reference block may consist of a smaller piece of test material in a larger, integral mount, or a deposited surface layer on a substrate. If this is the case, care must be taken by the test block manufacturer to ensure that the stiffness of that integral mount or substrate is sufficiently high that its compliance does not significantly affect the measured elastic properties of the test material.

A2.5 Certification Procedure

A2.5.1 The test block manufacturer shall determine both the dynamic Young's modulus and Poisson's ratio for the test block material, each to within 1.0 % accuracy, using the current versions of Test Methods E1875 or E1876. This process may be performed either on each block or on a larger batch of material prior to sectioning or separation of individual test blocks. If such "batch certification" is performed, the manufacturer shall confirm, by testing a limited but statistically significant number of specimens that blocks from various locations within the original batch all meet the general requirements given in A2.2.

A2.5.2 The test block manufacturer shall determine general guidelines concerning which indenter geometry's are suitable for each test block, and the force or depth range over which the blocks will perform satisfactorily. This information depends not only on the particular test block material, but on block preparation as well.

A2.5.3 The test block manufacturer shall confirm that the elastic properties of the block at its test surface do not deviate from the bulk values by more than 5 %, due to, for example, any grinding, polishing or annealing processes used in the preparation of the blocks. Methods to accomplish this could include, for example, test indentation by the manufacturer or measurement of surface elastic properties by surface acoustic wave methods.

A2.5.4 Surface roughness shall be measured on each polishing batch, or on each deposition batch, in the case of a deposited test surface layer. The manufacturer shall report the method used for surface roughness determination.

A2.5.5 Each block must be marked with it's own a serial number or letters. The markings may be on the top or side of the block. If the marking is on the side of the block, the markings shall be upright when the test surface is the upper surface.

A2.6 Certification Report

A2.6.1 The report for each test block shall at contain the following minimum information:

A2.6.1.1 The name of the laboratory certifying the block,

A2.6.1.2 The certified values for dynamic Young's modulus, *E*, and Poisson's ratio, ν, along with the uncertainty of each,

A2.6.1.3 The method by which *E* and ν were determined, including identification of the equipment used and relevant traceability information for that method and equipment,

A2.6.1.4 The serial number of the block,

A2.6.1.5 The date of certification,

A2.6.1.6 The regions of the block surface that is not available to the user for indentation; examples of such regions include areas that are too close to an edge, or regions that were used by the manufacturer for indentation or other quality control testing that might have altered the surface properties,

A2.6.1.7 The indenter geometries for which the block is appropriate, and range of indentation force or depth over which the block may be expected to perform satisfactorily for each specified indenter geometry,

A2.6.1.8 The surface roughness of the test surface, including a precise definition of the roughness quantity reported and a description of how it was determined, and

A2.6.1.9 An expiration date, if one is appropriate for a given test material.

A3. INDENTER REQUIREMENTS

A3.1 Scope

A3.1.1 This annex will define the requirements for the various indenters typically used for IIT. The physical dimensions and manufacturing tolerances of the most common indenters will be defined along with the requirements for certification.

A3.2 General Requirements

A3.2.1 The indenters used for IIT can be many different shapes to suit the test method used. All indenters shall meet the following requirements:

A3.2.1.1 The part of the indenter that contacts the sample shall be made from a hard material and have a defined shape. They can be a one piece or multi-piece design.

A3.2.1.2 The surface of the indenter that contacts the sample shall be highly polished and free from chips, pits, contamination and any other imperfections that may affect its final use. The surface shall be observed under a microscope with a magnification of least 50×.

Note A3.1—Spherical indenters that meet the requirements of ABMA/ISO 3290-1 Grade 24 do not have to be inspected optically.

A3.2.1.3 Each indenter shall have a unique serial number. In the case of a ball indenter, the holder only shall be serialized. The serial number shall be marked on the indenter or holder in a manner that cannot be easily removed. Indenters that are too small to be easily marked shall have the serial number marked on its container.

A3.2.1.4 The indenters shall be measured to verify their conformance to the dimensional requirements. A nominal indenter area function at the maximum usable indentation depth shall be calculated based on the actual dimensions of the indenter.

A3.2.1.5 Spherical indenters that are part of a batch of balls from a lot that meet the requirements of ABMA/ISO 3290-1 Grade 24 do not have to be individually measured or have a indenter area function determined.

A3.2.1.6 Indenters for use at indentation depths ≤0.006 mm shall have their area function defined over the relevant indentation depth range of use per A3.7.

A3.3 Vickers Indenters

A3.3.1 Vickers indenters that are used for IIT are similar to the indenters defined in Test Methods E384 and E92. They shall meet the following requirements:

A3.3.1.1 The angle between the opposite faces of the vertex of the diamond pyramid shall be (136 ± 0.3°) (see Fig. A3.1).

A3.3.1.2 The angle between the axis of the diamond pyramid and the axis of the indenter holder (normal to the seating surface) shall not exceed 0.5°.

A3.3.1.3 The four faces should meet at a sharp point. The maximum permissible length of the line of conjunction, c, between opposite faces shall be 0.001 mm (see Fig. A3.2).
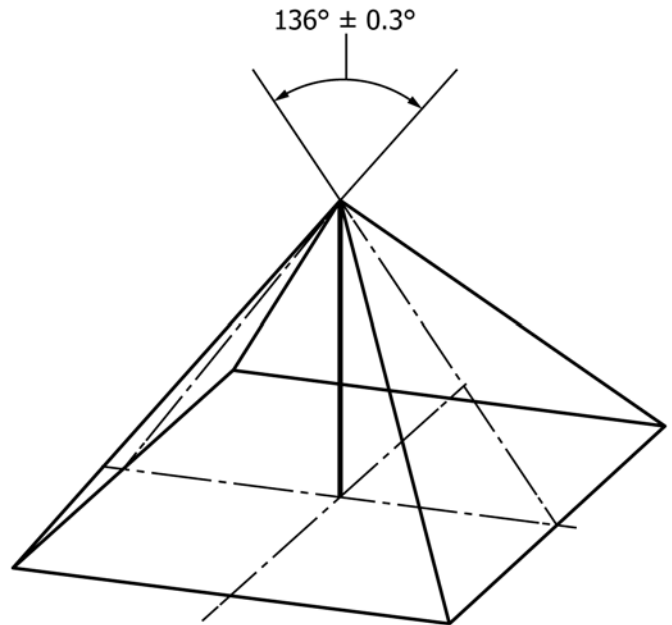

FIG. A3.1 Angle of the Vickers Diamond Pyramid

A3.4 Three Sided Pyramidal Pointed Indenters

A3.4.1 There are three commonly used three sided pyramidal indenters used for IIT. They shall meet the following requirements:

A3.4.1.1 *Berkovich and Modified Berkovich*—There are two types of Berkovich pyramidal diamond indenters in use. The original Berkovich indenter was designed to have the same surface area as a Vickers indenter at any given indentation depth. The modified Berkovich indenter is more commonly used and has the same projected area as a Vickers indenter at any given indentation depth. The angles and tolerances for a Berkovich indenter shall meet the angle and tolerance requirements defined in Fig. A3.3.

A3.4.1.2 *Cube Corner*—The angles and tolerances for a cube corner indenter shall meet the angle and tolerance requirements defined in Fig. A3.3.

A3.5 Spherical Ball Indenters

A3.5.1 The ball shall be harder than the test piece. Carbide balls with hardness not less than 1500 HV10 and having the chemical composition defined in Table A3.1 are recommended.

A3.5.2 The balls shall meet the tolerance defined in Table A3.2. It is permissible to certify their compliance to the requirements of this section by using batch inspection techniques.

Note A3.2—Balls that conform to ABMA/ISO 3290-1 Grade 24 satisfy these requirements.

A3.6 Spherical Tipped Conical Indenters

A3.6.1 Indenters with a spherical tipped cone shape are useful for many applications. These indenters are normally made from diamond but may also be made from other
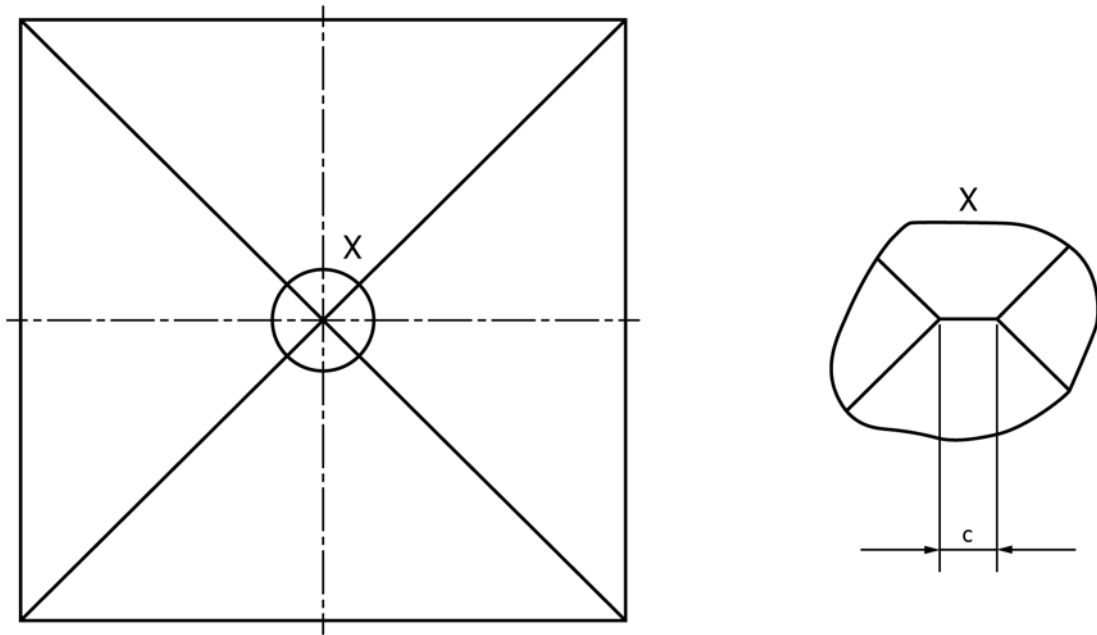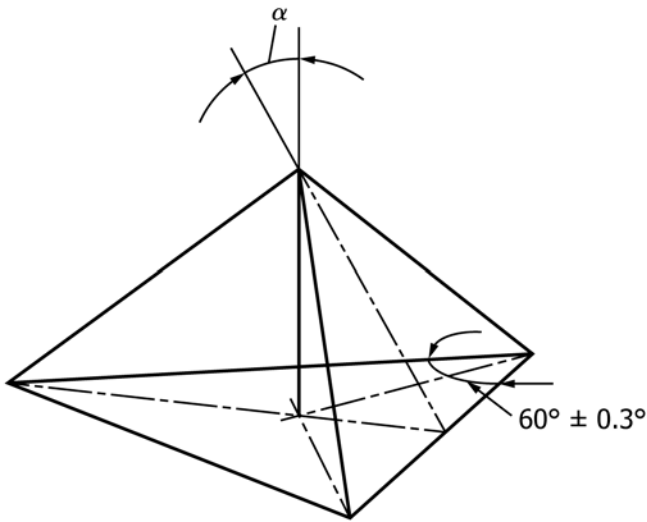
FIG. A3.2 Line of Conjunction at the Tip of the Indenter, Schematically

α = 65.03° ± 0.3° for Berkovich indenter
α = 65.27° ± 0.3° for modified Berkovich indenter
α = 35.26° ± 0.3° for corner cube indenters
FIG. A3.3 Angle of the Berkovich and Cube Corner Indenters

TABLE A3.1 Carbide Ball Chemical Composition

Chemical	Percent
Cobalt (Co)	5.0 to 7.0 %
Total other carbides	2.0 %
Tungsten Carbide (WC)	balance

TABLE A3.2 Tolerances for Ball Indenters

Ball Indenter Diameter, mm	Tolerance, mm
10	±0.005
5	±0.004
2.5	±0.003
1	±0.003
0.5	±0.003

TABLE A3.3 Tolerances for Sphero-Conical Indenters

Feature	Tolerance
Average Radius (R_{av}) $\leq$ 0.050 mm	±0.25 R_{av}
0.500 > R_{av} > 0.050 mm	±0.10 R_{av}
Cone included angle (2α)	
120°	±5°
90°	±5°
60°	±5°
Cone flank angle (α) to centerline of mount	
60°	±5°
45°	±2.5°
30°	±2.5°
Point of intersection of cone flanks to centerline of mount	within 0.01 mm

materials, for example, ruby, sapphire or hard metal as long as the material is significantly harder than the sample being tested. They are intended to indent only with the spherical tip. The characteristics of spherical tipped conical indenters shall be as given in Table A3.3.

A3.6.2 The instantaneous radius of curvature ($R(h)$) of the spherical cap at any indentation depth h measured from the point of first contact should not vary by more than a factor of two from the average radius, that is, $0.5 < R(h)/R_{av} < 2$.

NOTE A3.3—Geometry suggests that the depth of spherical cap h_s on a cone of included angle 2α and radius R_{av} is given by:

$$h_s = R_{av}(1 - \sin(\alpha)) \qquad (A3.1)$$

A3.6.3 In practice, there is a gradual transition from spherical cap to cone geometry, which is hard to specify. Given this and the uncertainties in R_{av} and α allowed (see Table A3.3), caution should be exercised whenever the depth exceeds $0.5\,h_s$.

A3.7 Indenter Area Function

A3.7.1 Most of the results determined from an IIT test are based on the projected contact area of the indenter. However,

usually, only the indentation depth is measured. When the maximum contact depth, h_c, is less than 6 µm, the relationship between depth and projected contact area may be significantly different from that predicted by the nominal area function. Therefore, when the indenter is used in this regime, a refined area function shall be determined. Either of the following techniques is recommended:

A3.7.1.1 A direct measurement method using a traceable atomic force microscope (AFM).

A3.7.1.2 Indirectly by utilizing indentations into a material of known Young's modulus (see Appendix X3).

A3.8 Report

A3.8.1 At least the following items shall be included in the report:

A3.8.1.1 Date of verification,

A3.8.1.2 Verifying laboratory,

A3.8.1.3 Description of indenter,

A3.8.1.4 Reference to this practice,

A3.8.1.5 Unique serial number,

A3.8.1.6 Geometrical data with an uncertainty statement,

A3.8.1.7 Nominal area function and maximum valid depth,

A3.8.1.8 Refined area function (if determined) and valid depth range, and

A3.8.1.9 Description of technique used to determine refined area function (if determined).

APPENDIXES

(Nonmandatory Information)

X1. ZERO-POINT DETERMINATION

X1.1 Scope

X1.1.1 This appendix describes two analyses by which one may determine the zero point for an individual instrumented indentation test and the corresponding uncertainties in force and displacement. Because this practice pertains only to quasistatic instrumented indentation testing of time-invariant materials, determination of zero-point with respect to time is not essential and is not addressed. Assigning $t = 0$ to the first (F, h, t) triple is common practice.

X1.1.2 The analyses in this appendix are intended to be used post-test to assign the zero point for the purpose of data analysis. For a variety of reasons, it may be necessary to assign a coarse zero point during the actual experiment.

X1.1.3 Some comments about the uncertainty of the zero point at very low forces are given in X1.4.

X1.2 Procedure A

X1.2.1 This procedure may be applied if both force and displacement are acquired continuously throughout approach and contact. It is not appropriate for systems, which apply a specific preload before initiating acquisition of force, displacement, and time data. It should be noted at the outset that this procedure calls for calculating new data series based on the original force data. *These series are only for the purpose of determining the zero point.* The original force data series will be denoted F_0. Subsequent series are denote with incremented subscripts: F_1, F_2, and F_3.

X1.2.2 Create F_1 by adjusting F_0 so that pre-contact forces are centered about zero. At least two options are available for accomplishing this task: compensation with a known analytic function or repeated differentiation. As an example of the compensation option, a load cell may have some known offset of 5 mN, and so force readings before contact are scattered about that value. Consequently, all F_0 values should be reduced by 5 mN. As a second example, there may be a linear relationship between force and displacement before contact due to the stiffness of the testing instrument, as revealed by a linear fit to the pre-contact data. If that is the case, then all F_0 values may be reduced by the amount $(mx + b)$, where m and b are the slope and intercept of the linear fit, and x is the displacement data.

X1.2.3 Now let us consider the option of repeated differentiation. For the example of the load cell with an offset of 5 mN, the first derivative of force (F_0) with respect to displacement will yield pre-contact force data that are centered about zero. For the example of a testing instrument with a linear relationship between force and displacement before contact, the second derivative will produce the same result. It should be noted that differentiation may be accomplished by several means, including numerical and electronic. The advantage of repeated differentiation is that it can be done with no *a priori* information regarding the pre-contact functional relationship between force and displacement. (Or more simply, one doesn't need to know the stiffness and offset of the testing instrument.) The disadvantage is that repeated numerical differentiation of experimental data tends to produce series that are progressively more "scattered."

X1.2.4 *Optional*—Create F_2 by summing F_1 over a sliding window of N points. Although this step is optional, it can significantly increase the sensitivity to contact because gradual changes from the baseline (zero) are accumulated. The first $N–1$ entries in F_2 are invalid. The Nth value for F_2 is calculated by summing the first N values of F_1. The $(N+1)^{st}$ value for F_2 is calculated by summing the second through the $(N+1)^{st}$ values of F_1, and so forth. Note that N should be large enough to cover several cycles if there is any periodicity in the pre-contact data. (A single value of N should be chosen for a particular combination of instrument and data acquisition rate; that is, once chosen, N should not change frequently.)

X1.2.5 Calculate the standard deviation in F_1 (or F_2 if created) before contact, σ.

X1.2.6 Create F_3 by dividing F_1 (or F_2 if created) by σ. It should be noted that the series F_3 is dimensionless and has the meaning of multiples of the pre-contact noise (as quantified by the standard deviation) in either F_1 or F_2.

X1.2.7 Tag the data point S_2 to be the first point for which F_3 exceeds 10.

X1.2.8 Tag the data point S_1 to be the last point before S_2 for which F_3 is less than 1.

X1.2.9 Set the zero point, S, to be the first point after S_1 for which F_3 is greater than 4.

Note X1.1—S_2, S_1, and S may all be interpolated points.

X1.2.10 Calculate the uncertainty in force due to the uncertainty in the assignment of the zero point to be half the change in F_0 between S_1 and S_2.

X1.2.11 Calculate the uncertainty in displacement due to the uncertainty in the assignment of the zero point to be half the change in displacement between S_1 and S_2.

X1.2.12 *Example of Using Procedure A*—Table X1.1 provides force-displacement data through approach and contact between a diamond indenter and polycarbonate. The following steps are used to determine the zero point and the associated uncertainties with respect to both force and displacement.

X1.2.12.1 *Step A1*—Using the first 108 data points (data up to –20 nm), the force-displacement data are fit to a line. This yields a slope of –0.00409 and an intercept of 0.17962. Using this line, the series F_1 is calculated so that pre-contact force values are about zero. These compensated data are plotted in Fig. X1.1.

X1.2.12.2 *Step A2*—The series F_2 is created using a window size of 20 data points. The first 19 entries in F_2 are invalid. The 20th entry in F_2 is the sum of the first 20 values of F_1. The 21st entry in F_2 is the sum of values 2 ~ 21 of F_1, and so forth.

X1.2.12.3 *Step A3*—The standard deviation in F_2 is calculated over the first 108 points to be 0.1045 µN.

X1.2.12.4 *Step A4*—The series F_3 is created by dividing all values in the series F_2 by 0.1045 µN.

X1.2.12.5 *Step A5*— S_2, S_1, and S are all determined as described in the procedure and plotted in Fig. X1.1.

X1.2.12.6 *Step A6*—The uncertainties with respect to force and displacement due to the assignment of the zero point are calculated to be ±0.085 µN, and ±0.81 nm, respectively.

X1.3 Procedure B

X1.3.1 Indenter tips generally possess a rounded apex that can be described geometrically as a sphere or as a paraboloid
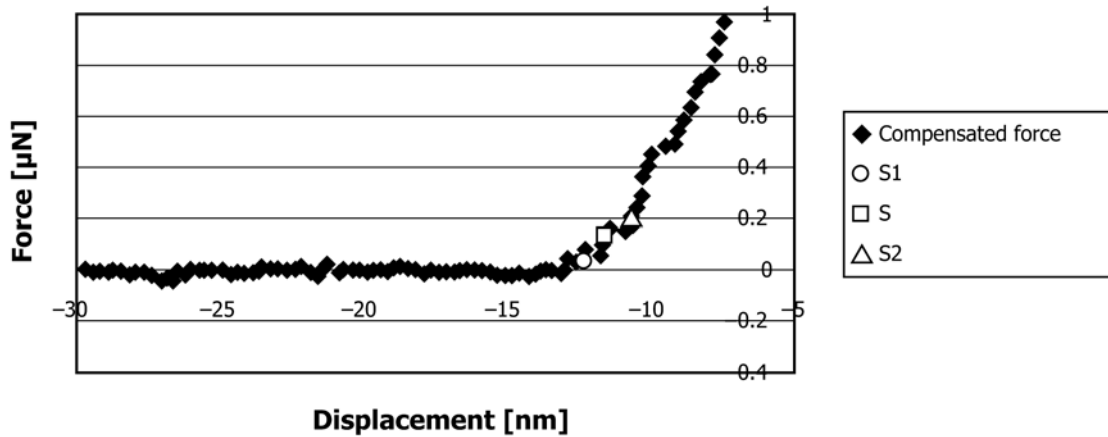
of revolution. Consequently, the initial portion of the loading curve often follows the contact mechanics originally formulated by Hertz, that is, $F = 4/3E_r R^{1/2}D^{3/2}$, where F is load, E_r is reduced modulus, R is combined radius of curvature (equal to the apical radius of curvature of the tip in the case of indentation into a flat specimen), and D is sum of tip and sample deformation. This equation is expected to hold true in the absence of appreciable attractive surfaces forces, up to the onset of plastic deformation, assuming that the shape function of the tip does not deviate from the rounded geometry over the range of wholly elastic deformation. A note of caution: the Hertz equation applies rigorously to the parabolic geometry but only approximately to the spherical geometry. Nevertheless, the spherical geometry is virtually identical to the parabolic geometry as long as the contact radius remains much less than the apical radius of curvature of the tip. Furthermore, nearly all materials yield before these two geometries deviate significantly.

X1.3.2 This procedure for eliminating the zero point error involves fitting the Hertz equation to the initial portion of the loading curve in order to determine the shift in displacement origin that minimizes the fit residuals. It is appropriate both for instruments that acquire pre-contact data and instruments that commence the load cycle from a small intentional preload. Because this procedure requires data within the range of wholly elastic deformation, the instrument must be capable of making measurements at very low contact stresses, through a combination of adequate force and displacement sensitivities and sufficiently large radius of curvature of the apex of the tip.

X1.3.3 The as-acquired load-displacement curve may possess a residual slope (for example, uncompensated spring force) and a residual offset (for example, constant offset in the load cell output) in its baseline. Identify a portion of the load-displacement curve known to represent out-of-contact data. This portion may correspond to pre-contact data or data obtained after fully extracting the tip from the specimen when completing the unloading process. Once identified, perform a linear curve fit to the selected out-of-contact data and subtract the resulting fit equation (of $y = mx + b$ form, where y is force and x is displacement) from the entire data set in order to obtain a new baseline that is statistically flat and without offset.

X1.3.4 Make a best-guess manual correction to the displacement zero point if it is obvious that a significant shift along the displacement axis would result in a substantially improved displacement zero point.

X1.3.5 Inspect the initial portion of the loading curve to identify the onset of plasticity. In some instances, the onset of plasticity is signaled by a displacement excursion (pop-in) in the case of load control instruments or a sudden load drop in the case of displacement control instruments. In other instances, the onset of plasticity is not so dramatic but a point where the Hertz equation no longer can hold true still may be evident. Use these observations as a guide for choosing the range of data to be included in the subsequent curve fitting procedure. If available, also include a segment of pre-contact and potentially pre-contact data leading up to the best-guess point of initial contact. Ideally, the chosen range of data is

TABLE X1.1 Indentation Depth at 1 µN for Various Combinations of Reduced Modulus (E_r) and Indenter Radius of Curvature as Estimated from Hertz Contact Theory

E_r (GPa)	Radius of Curvature of Indenter (nm)		
	10	100	1000
0.1	178	82.5	38.3
1	38.3	17.8	8.25
10	8.25	3.83	1.78
100	1.78	0.825	0.383

NOTE 1—Uncertainty in force is ±0.085 μN; uncertainty in displacement is ±0.81 nm.
FIG. X1.1 Determination of Zero Point (*S*) for a Test on Polycarbonate

comprised of a segment of pre-contact data followed by in-contact data leading up to the onset of plasticity.

X1.3.6 If the onset of plasticity is clearly evident, simply use a suitable nonlinear curve-fitting algorithm (for example, Levenberg-Marquardt) to fit the Hertz equation to the data range selected in step X1.3.5. For the purpose of curve fitting, the Hertz equation can be parameterized as $F = C(D+D_s)^{3/2}$, where C is a fit coefficient and D_s is a rigid displacement shift. A note of caution: the chosen algorithm must be able to handle domain errors that might arise from attempts to compute the 3/2 power of a negative number. The algorithm employed for the example given below involves defining a fitting window specified by left-side and right-side displacement cutoffs. The algorithm performs curve fitting iteratively, correcting the displacement values after each cycle by the newly found D_s, until D_s finally takes on a value of zero (or near zero as defined by a suitable convergence criterion). The total correction to the displacement zero point is the sum of D_s from each cycle and the manual correction made in step X1.3.4. The algorithm described here is effective at pulling the data to the correct position within the fitting window if two initial conditions are met:

X1.3.6.1 The first cycle is seeded by data clearly representing contact falling within the fitting window; and

X1.3.6.2 The position of the zero point after manual correction is in the vicinity of the fitting window.

X1.3.7 Repeating step X1.3.6 as a function of right-side displacement cutoff may be necessary if a range of data representing wholly elastic displacements is not evident. As a general observation, both C and D_s often remain flat over a range of right-side displacement cutoffs, but change rapidly when the right-side displacement cutoff is too small (owing to extremely ill-defined contact geometry, effects caused by chemical contaminants, influence of instrument noise, etc.) or too large (owing to the inclusion of data representing plastic deformation). A suitable choice for the right-side cutoff is one that falls within the range of stable C and D_s. It is noted that attempts to utilize the correlation coefficient as a metric for establishing the best choice of fitting window proved to be unsuccessful. Visual comparison clearly indicated that the correlation coefficient was impacted more by the number of points included within the fitting window than by the quality of fit over the range of included points.

X1.3.8 The algorithm described here estimates uncertainties in force and displacement zero points by computing root-mean-square errors from the comparison of the fully corrected experimental curve to the best-fit theoretical curve. Any fully corrected experimental point with load and displacement values at zero or positive are included in the uncertainty analysis, as long as it falls within an uncertainty radius defined as some fraction of the right-side displacement cutoff.

X1.3.9 *Example of Using Procedure B*—The load-displacement data (collected under load control) used in this example is identical to that of the example given in Procedure A.

X1.3.9.1 *Step B1*—The residual slope in the out-of-contact load signal is eliminated by subtracting the linear fit to the loading data, over the range of –47.1 to –15 nm, from the entire load-displacement curve.

X1.3.9.2 *Step B2*—It is evident that adding 10 nm to all displacement values would markedly improve the displacement zero point.

X1.3.9.3 *Step B3*—A fitting window with a left-side displacement cutoff of –5 nm and a right-side displacement cutoff of 10 nm is chosen for the Hertzian curve-fitting procedure. The best-fit D_s is positive 12.34 nm relative to the as-acquired load-displacement curve (compared to 11.61 nm for Procedure A). The uncertainties associated with the zero point are estimated as ±0.065 μN and ±0.43 nm (compared to ±0.085 μN and ±0.81 nm for Procedure A) for an uncertainty radius of 25 % of the right-side displacement cutoff (or 2.5 nm). Accounting for the uncertainties, Procedures A and B are in good agreement. It is noted, however, that Procedure B is more likely to suppress the impact of non-ideal behavior often observed at the shallowest of contact depths in comparison to Procedure A.
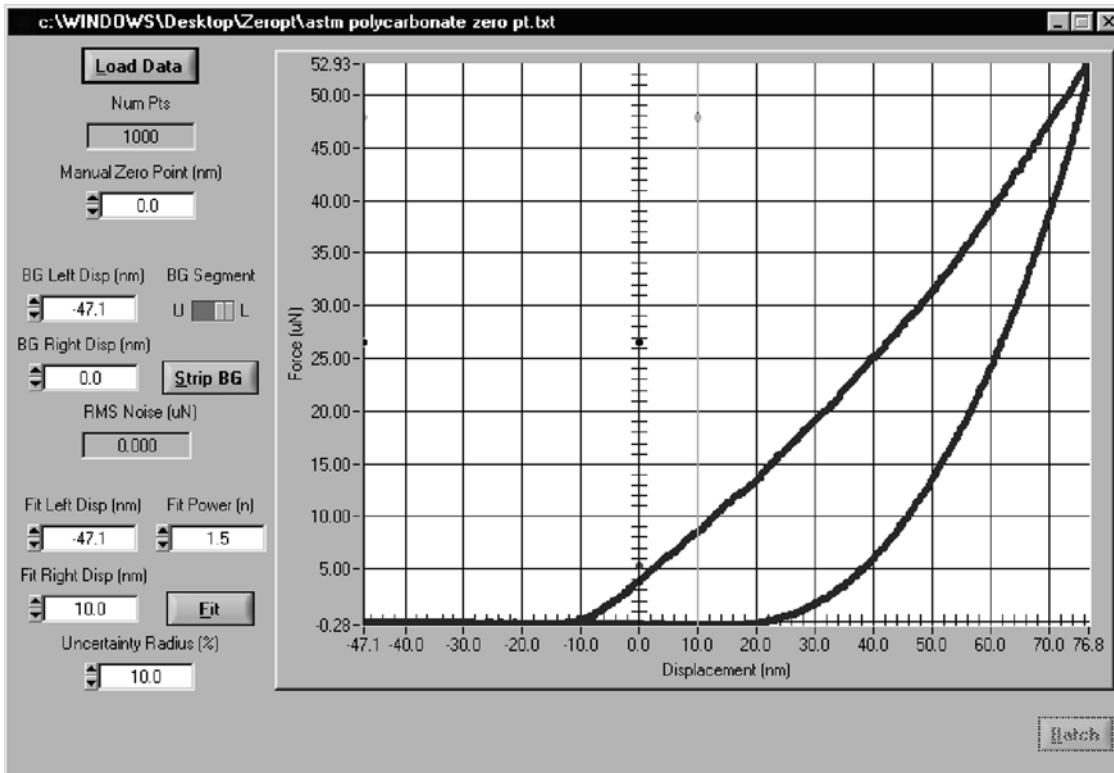
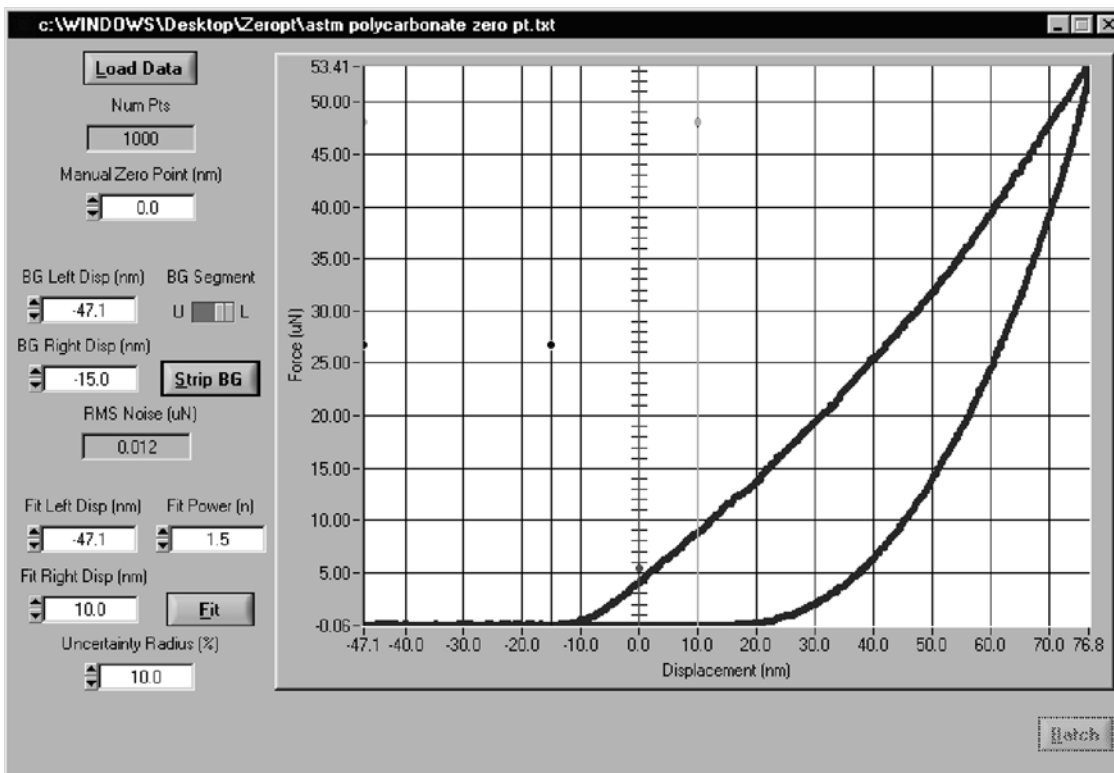**FIG. X1.2 As-acquired Force-Displacement Curve for Polycarbonate**

FIG. X1.3 Force-Displacement Curve After Correcting for Residual Slope in Out-of-Contact Load Signal

X1.3.9.4 *Step B4*—Because the onset of plasticity for polycarbonate did not involve a sudden displacement excursion, a more thorough fitting analysis was conducted by examining the evolution of best-fit D_s and C over a range of right-side
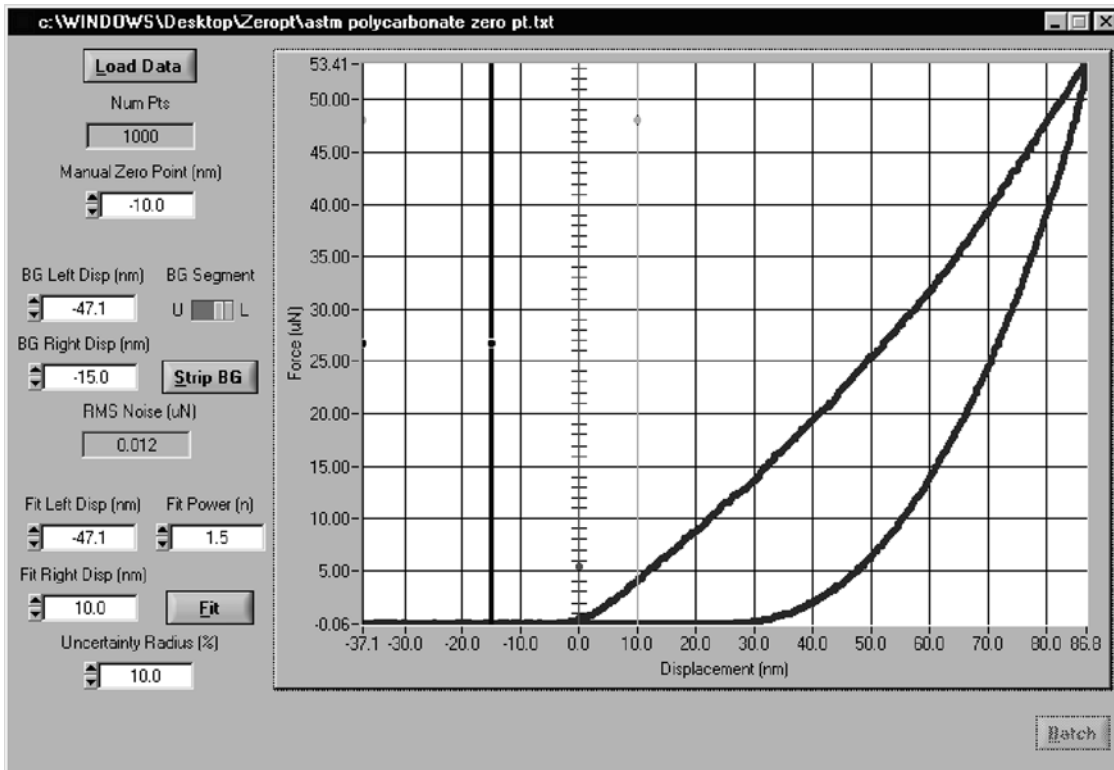
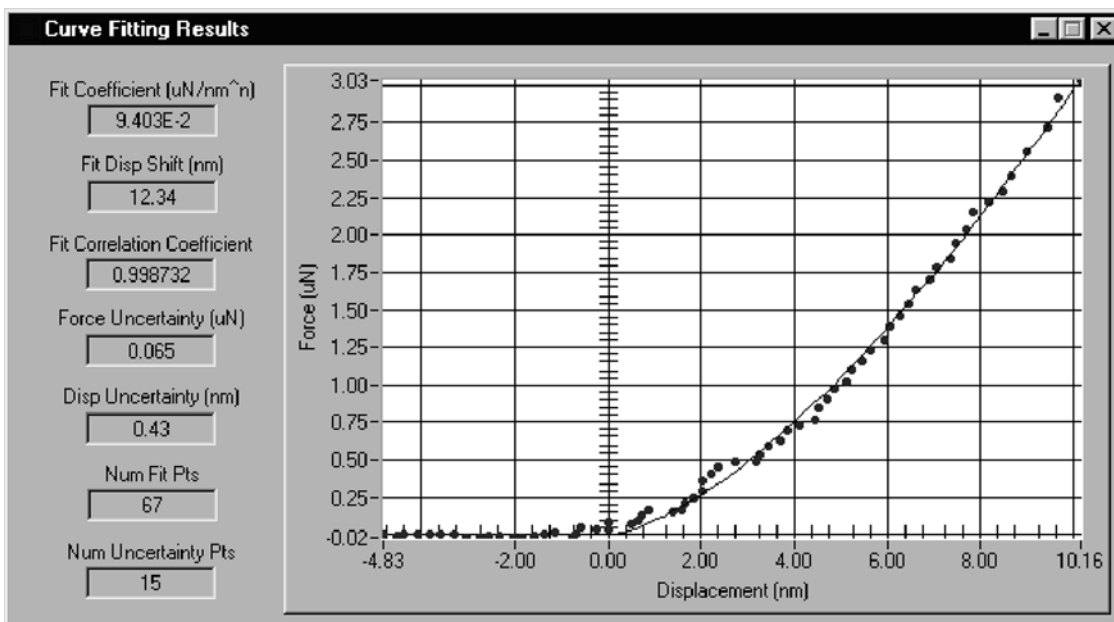FIG. X1.4 Force-Displacement Curve After Manual Shift of Zero Point

FIG. X1.5 Hertzian Fit to Force-Displacement Curve and Fitting Statistics

displacement cutoffs. Plots of these two parameters against right-side displacement cutoff indicate that the curve-fitting results of X1.3.9.3 are adequate for defining the zero point.

X1.4 Uncertainty

X1.4.1 For applications using very low test forces the uncertainty in the displacement at the zero point is highly dependent on force and displacement noise floors, force and displacement accuracies at very small values, the radius of curvature of the indenter, the elastic modulus of the test specimen, the presence of adsorbed water, other sources of attractive forces, etc. Table X1.1 displays estimates of indentation depth at 1 µN (a very low test force) for various combinations of reduced modulus and indenter radius of curvature. As can be seen from the table, a compliant sample in combination with a sharp indenter could result in a large

15

polycarb left neg5
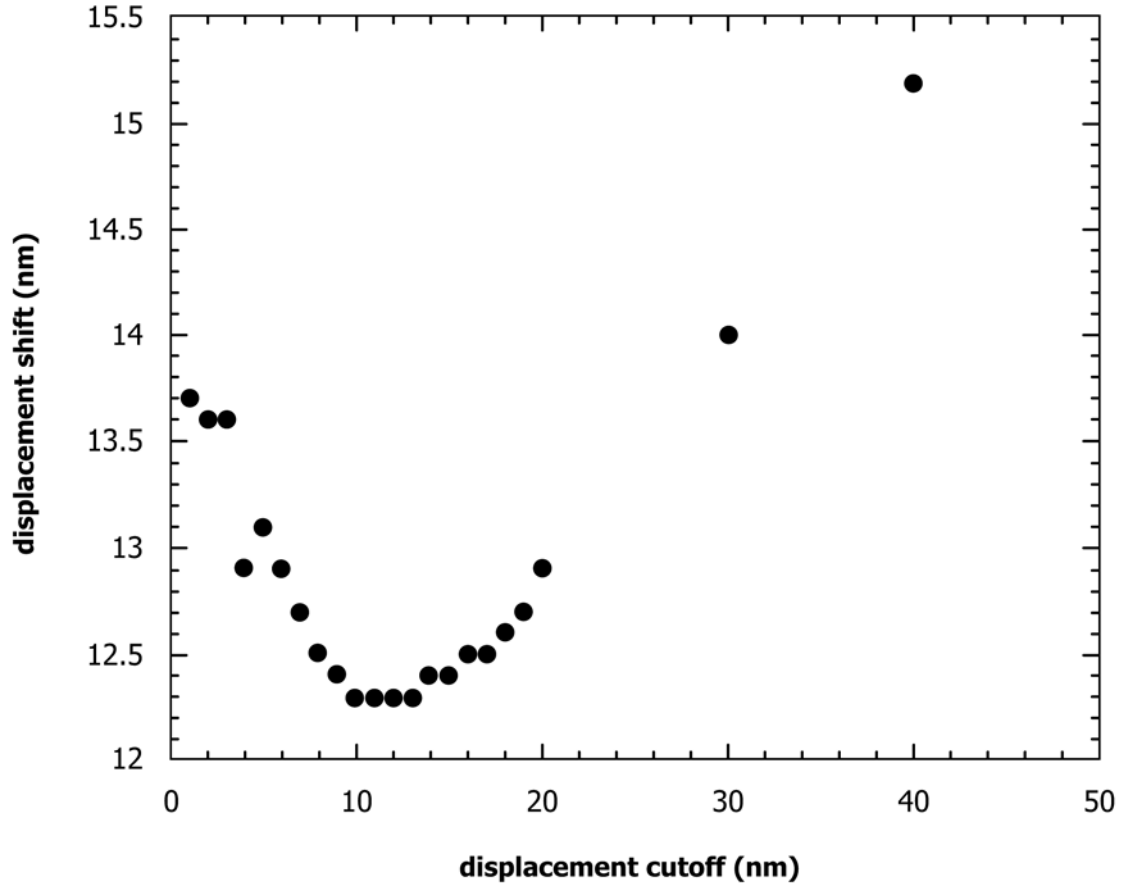
NOTE 1—Region of low gradient (10 nm [D_s] 13 nm) indicates range of stable curve fits with respect to Hertz equation.
FIG. X1.6 Best-Fit D_s for Various Right-Side Displacement Cutoffs, with Left-Side Displacement Cutoff Fixed at –5 nm

absolute uncertainty in the displacement at the zero point if the force noise floor of the instrument is not substantially better than 1 µN.

polycarb left neg5
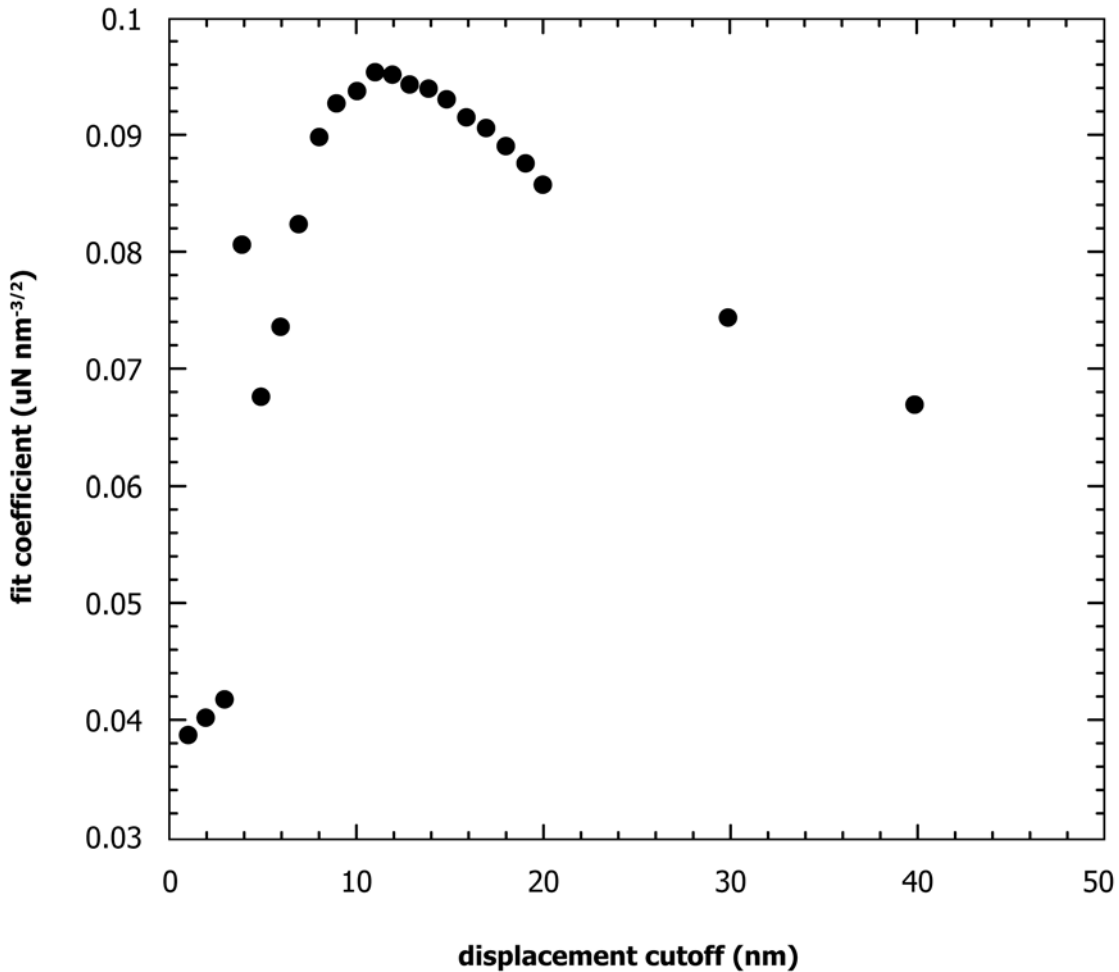
NOTE 1—Region of low gradient is consistent with range of stable values of D_s found in Fig. X1.5.
FIG. X1.7 Best-Fit *C* for Various Right-Side Displacement Cutoffs, with Left-Side Displacement Cutoff Fixed at –5 nm

X2. DETERMINATION OF INSTRUMENT COMPLIANCE

X2.1 Scope

X2.1.1 This appendix describes a procedure by which instrument compliance may be determined for the instrument described in Section 6 of this practice.

X2.2 Referenced Documents

X2.2.1 Numerical Recipes in FORTRAN: The Art of Scientific Computing, Second Edition, William H. Press, Saul A. Teukolsky, William T. Vetterling, Brian P. Flannery, ISBN 0-521-4306-X, Cambridge University Press, 1992.

X2.3 Definitions

X2.3.1 δ = indenter displacement into surface beyond which indentation modulus is expected to be constant for a particular combination of indenter and SRM.

C_f^* = value of instrument compliance used to calculate results according to Appendix X4 of this practice (may be zero).

C_Δ = *correction* to original value of C_f^*.
C_f = new value for instrument compliance.

X2.4 General Conditions

X2.4.1 The instrument and procedure shall comply with Sections 6 and 8 of this practice, respectively. Any indenter that meets the requirements of Annex A3 may be used for this procedure.

X2.5 Test Sample

X2.5.1 This procedure requires the use of a standard reference block as described in Annex A2.

NOTE X2.1—If the indentation procedure produces any fracture in the test material, the data shall not be used for this analysis. The indentation should be imaged to determine if fractures have occurred. An alternate standard reference material must be selected if fractures occur.

NOTE X2.2—The analysis relies on the assumption that when the indenter displacement into the surface, h, is greater than some value, δ, the indentation modulus is constant (although not necessarily known) for all

greater penetration depths.

X2.6 Procedure

X2.6.1 The test method defined in Appendix X4 is used to perform a series of tests on the standard reference block in which the maximum indenter displacement into the surface is varied over the range of depths to be included in the determination of instrument compliance. The minimum depth to be included is limited by δ for the particular combination of test block and indenter used. Barring any physical limitations of the test block, such as cracking or block thickness, the maximum depth should be limited by the force or displacement limit of the instrument, because the best estimate of instrument compliance is obtained for large indentations. Also, the reliability of the resulting instrument compliance increases with the number of individual tests used in its determination.

X2.7 Analysis

X2.7.1 Test method in Appendix X4 requires that the data be fully corrected for instrument compliance; thus an original value of instrument compliance C_f^* must be used, even if that value is assumed to be zero (no instrument compliance). All calculations described in this appendix use the results calculated according to Appendix X4 using the original estimate of instrument compliance, C_f^*. The new value for instrument compliance, C_f, is determined by:

$$C_f = C_f^* + C_\Delta \tag{X2.1}$$

where:

C_Δ = *correction* to C_f^*.

X2.7.2 In addition to the analysis required by Appendix X4, determination of C_Δ requires calculation of the quantity C_s for each test using:

$$C_s = 1/S \tag{X2.2}$$

X2.7.3 Thus, each indentation test yields a value of C_s and a value of A_p (projected contact area at F_{max}). The series of (A_p, C_s) pairs for all tests are fit to the linear form:

$$C_s = m(A_p^{-1/2}) + C_\Delta \tag{X2.3}$$

where:

m and C_Δ = best-fit constants. (See "Fitting Straight Line to Data," X2.2.)

X2.7.4 Once C_Δ is known, C_f can be calculated directly.

X2.7.5 Alternatively, if it may be reasonably assumed that *both* the indentation hardness and modulus should be uniform for all indentation depths used in this procedure, then the following analysis may be used to determine C_Δ.

NOTE X2.3—This is usually a good assumption for Berkovich and Vickers indenters; it is usually *not* a good assumption for spherical-tipped indenters.

X2.7.6 This analysis is advantageous because it does not require determination of the projected contact area, A_p.

X2.7.7 The series of (F_{max}, C_s^*) pairs for all tests are fit to the linear form:

$$C_s^* = m(F_{max}^{-1/2}) + C_\Delta \tag{X2.4}$$

where:

m and C_Δ = best-fit constants.

X3. INDIRECT DETERMINATION OF INDENTER REFINED AREA FUNCTION

X3.1 Scope

X3.1.1 This appendix describes a procedure by which one may determine a refined area function for a particular indenter as required in A3.7.

X3.2 Apparatus

X3.2.1 The instrument used for this determination shall meet all the requirements of Sections 6 and 8 of this practice.

X3.3 Procedure

X3.3.1 This procedure requires the use of a standard reference block as described in Annex A2. In particular, the material fused silica is recommended because it is elastically isotropic, does not oxidize, and material does not tend to pile up around the indenter during testing. This procedure uses the known reduced modulus for the standard reference block, E_r.

X3.3.2 The test method defined in Appendix X4 is used to perform a series of tests on the standard reference block in which the maximum indenter displacement into the surface is varied over the range of depths to be included in the determination of the refined area function. The range of depths to be included is typically limited by resolution and range of the instrument. The reliability of the refined area function increases with the number of individual tests used in its deter-

mination. In addition a heavier concentration of tests at the lower depths is important. When an indenter area function is called for by the analysis, the nominal indenter area function is used, since the refined area function has not yet been determined.

X3.4 Analysis

X3.4.1 In addition to the analysis required by the test method, the quantity A_E is also calculated for each test using:

$$A_E = \pi S^2 / 4 E_r^2 \tag{X3.1}$$

where:

E_r = known reduced modulus for the standard reference block, not the value computed as a result of the analysis required by Appendix X4.

X3.4.1.1 Thus, each indentation test yields a value of A_E and a value of h_c. The series of A_E and h_c pairs for all tests are fit to the functional form:

$$A_E = C_1 h_c^2 + C_2 h_c + C_3 h_c^{1/2} + C_4 h_c^{1/4} + C_5 h_c^{1/8} + \dots + C_n h_c^{1/n} \tag{X3.2}$$

where:

C_i = best-fit constants.

X3.4.2 For sharp indenters, C_1 should be restricted to that used in the nominal area function. For ball indenters or spherical-tipped conical indenters, C_2 should be restricted to that used in the nominal area function. It is further recommended that all coefficients, C_i, be limited to positive values. If these recommendations are followed, the refined area function is valid at depths greater than those used in the fit (up to the limit of validity of the nominal area function). However, it is sometimes difficult to obtain a good fit to the (h_c, A_E) pairs with these restrictions. If this is the case, a better fit may be obtained if all C_i are allowed to float to their best-fit values and be either positive or negative. In this case, the refined area function is only valid over the range of h_c included in the fit.

X3.4.3 This fitting procedure yields the refined area function for the diamond, $\Lambda(d)$ given by:

$$\Lambda(d) = C_1 d^2 + C_2 d + C_3 d^{1/2} + C_4 d^{1/4} + C_5 d^{1/8} + \ldots + C_n d^{1/n}$$
(X3.3)

where:

d = distance from the apex of the tip as measured along the central axis, and

Λ = projected (cross-section) area of the indenter tip at d.

X3.4.3.1 A good technique for evaluating the quality of the fit is to compute the normalized variation between the fit at each value of h_c and the corresponding value of A_E:

$$Var = (\Lambda(h_c) - A_E)/A_E$$
(X3.4)

X3.4.3.2 It is further recommended that the *Var* quantities be plotted as a function of h_c. It is desirable to see the *Var* quantities scattered about zero. The magnitude by which *Var* deviates from zero at a particular value of h_c is the relative error in A_p one may expect to incur when using the refined area function at that depth. The magnitude of this relative error may determine the lower depth limit for the valid range of the refined area function.

X4. TEST METHOD FOR MEASURING INDENTATION MODULUS AND HARDNESS USING INSTRUMENTED INDENTATION TECHNIQUES

X4.1 Scope

X4.1.1 This method is intended to provide a procedure and analysis by which one may determine the indentation modulus (E_{IT}) and indentation hardness (H_{IT}) of materials using instrumented indentation techniques as defined in this practice.

X4.1.2 SI units will be used for all parameters.

X4.1.3 *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.*

X4.2 Referenced Documents

X4.2.1 Numerical Recipes in FORTRAN: The Art of Scientific Computing, Second Edition, William H. Press, Saul A. Teukolsky, William T. Vetterling, Brian P. Flannery, ISBN 0-521-4306-X, Cambridge University Press, 1992.

X4.3 Terminology

X4.3.1 *pile-up*—a buildup of material around the edge of an indent that is the result of the indentation process.

X4.3.2 *sink-in*—a depression around the edge of an indent that is the result of the indentation process.

X4.3.3 *indentation modulus (E_{IT})*—the materials modulus determined from the unload curve of an indentation test performed according to this practice.

X4.3.3.1 *Discussion*—Indentation modulus (E_{IT}) is comparable to Young's modulus (E) when the projected contact area (A_p) can be accurately determined.

X4.3.4 *indentation hardness (H_{IT})*—the materials resistance to penetration, defined as the maximum test force divided by the projected area of the indenter at the depth of penetration.

X4.4 Summary of Test Method

X4.4.1 A hard indenter of known shape and elastic properties is slowly forced into the surface of a test sample and withdrawn. During the entire process the force applied to the indenter and the displacement of the indenter's tip into the surface of the material are recorded at specific time intervals such that a force-displacement curve could be generated. The indentation modulus (E_{IT}) is calculated from the slope of the unload curve. The indentation hardness (H_{IT}) is calculated by dividing the maximum force applied by the projected contact area at that maximum force.

X4.5 Significance and Use

X4.5.1 This method is intended to provide a procedure and analysis by which the indentation modulus (E_{IT}) and indentation hardness (H_{IT}) of a material can be determined using an instrumented indentation tester. These tests can be useful for evaluating thin coatings, specific phases, constituents too small for other types of mechanical tests and bulk materials. While the test sample may be altered at the indentation location, the technique is generally considered to be nondestructive.

X4.5.2 Indentation modulus (E_{IT}) is comparable to Young's modulus (E) when the projected contact area (A_p) can be accurately determined. The piling up or sinking in of material around the perimeter of the indentation may adversely affect the accurate determination of the projected contact area.

X4.5.3 Indentation hardness (H_{IT}) is very useful for materials evaluation, quality control of manufacturing processes, and research and development efforts. Hardness, although empirical in nature, can be correlated to tensile strength for many metals, and is an indicator of wear resistance and ductility.

X4.5.4 This method may be used to make the measurements required for indirect verification of an instrumented indentation machine (see Annex A1).

NOTE X4.1—This method is not recommended for testing materials that exhibit a significant time-dependent response to an applied stress.

X4.6 Apparatus

X4.6.1 The instrument used for this test method shall meet all of the requirements defined in Sections 6 and 8 of this practice.

X4.7 Test Sample

X4.7.1 The test sample shall conform to the requirements of Section 7 of this practice.

X4.8 Procedure

X4.8.1 *Configure Instrument:*

X4.8.1.1 *Indenter*—Any of the following indenter types may be used: Vickers, Berkovich, ball, or spherical-tipped cone. The indenter shall comply with the requirements of Annex A3.

X4.8.1.2 *Maximum Test Force*—Generally, the user should choose the largest force that will allow an accurate and meaningful evaluation of the sample. The following should be considered when choosing the maximum test force: sample thickness (especially when the sample is a film or coating), machine compliance, surface finish, and tendency for fracture.

X4.8.1.3 *Test Cycle*—Once the indenter contacts the surface, the indenter shall be pressed into the test surface in a smooth, controlled manner such that the maximum desired force or displacement is reached in 15 seconds. It is recommended that the force on the sample be held constant at the maximum value for 10 seconds. The indenter shall be withdrawn from the test surface in a smooth, controlled manner such that the test force is reduced to 10 % or less of the maximum test force in 15 seconds.

NOTE X4.2—Other times for loading, dwell and unload may be used, as long as such deviations are described in the test report and included in the designation of properties as described in Section 3.

NOTE X4.3—For shallow depth indents it may be desirable to have a constant load segment that is small relative to the maximum load to evaluate displacements due to drift.

X4.8.2 *Perform Test Cycle*—Perform the test in accordance with the requirements of Section 8 and the setup requirements indicated in section 8.1.

X4.8.3 *Perform Multiple Tests*—Five tests should be performed on each sample or set of samples of the same material if the test area is too small.

X4.8.4 *Calculate Results*—The following calculations shall be performed using a set of fully corrected force (F), displacement (h), and time (t) data for an individual test. (See section 6.3.2 of this practice.)

X4.8.4.1 *Indentation Modulus*—The indentation modulus is calculated as:

$$E_{IT} = (1 - v_s^2)\left[\frac{1}{E_r} - \frac{(1 - v_i^2)}{E_i}\right]^{-1} \quad (X4.1)$$

where:

v_s = Poisson ratio of the sample, and
E_i = Young's modulus of the indenter, and
v_i = Poisson ratio of the indenter.

(1) For diamond indenters, the values for E_i and v_i are 1140 GPa and 0.07 respectively. The reduced modulus E_r is calculated as:

$$E_r = (\pi/A_p)^{1/2} (S/2) \quad (X4.2)$$

(2) The contact stiffness (S) is determined by fitting the first 50-90 % of the force-displacement data (F-h data) acquired during unload to the power-law relation:

$$F = B(h - h_f)^m \quad (X4.3)$$

where:

B, m, and h_f = best-fit constants.

(3) The Levenberg-Marquardt method may be used to obtain these best-fit constants. (An explanation of this method and FORTAN code may be found in X4.2.) The contact stiffness, S, is calculated by analytically differentiating the power-law fit and evaluating at the maximum indenter displacement:

$$S = Bm(h_{max} - h_f)^{m-1} \quad (X4.4)$$

(4) Contact area is calculated by evaluating the indenter area function at h_c:

$$A_p = \Lambda(h_c) \quad (X4.5)$$

(5) The contact depth, h_c, is calculated using:

$$h_c = h_{max} - 3F_{max}/4S \quad (X4.6)$$

X4.8.4.2 *Indentation Hardness*—The indentation hardness, H_{IT}, is calculated as:

$$H_{IT} = F_{max}/A_p \quad (X4.7)$$

X4.8.4.3 *Example Calculations of E_{IT} and H_{IT}:*

(1) As an example, the force-displacement data shown in Fig. X4.1 resulted from a single indentation test cycle. The test material was fused silica (amorphous quartz) and the indenter was a modified Berkovich diamond. (Note: these data have been fully compensated according to the considerations provided in section 6.3.2.) The nominal elastic properties for the contacting materials are: Young's modulus (E_s) = 72 GPa, Poisson's ratio (v_s) = 0.18 for the fused silica, and Young's modulus (E_i) = 1140 GPa, Poisson's ratio (v_i) = 0.07 for the diamond. Intermediate and final results of this analysis are provided in Table X4.1.

(2) The first 30 of 60 unload data points (see Table X4.2) were curve fit to the form of Eq X4.3. Using a Levenberg-Marquardt algorithm, the best-fit constants, B, h_f, and m were determined. With h_{max} = 2161.93 nm, these values were used to calculate the stiffness, S, according to Eq X4.4 as:

$$S = 1.297*0.05786 \text{ mN/nm}^{1.279}*(2161.9 \text{ nm} - 1060.5 \text{ nm})^{1.297-1}$$
$$= 0.5999 \text{ mN/nm} \quad (X4.8)$$

(3) With F_{max} = 509.9 mN, the contact depth, h_c, was calculated according to Eq X4.6 as:

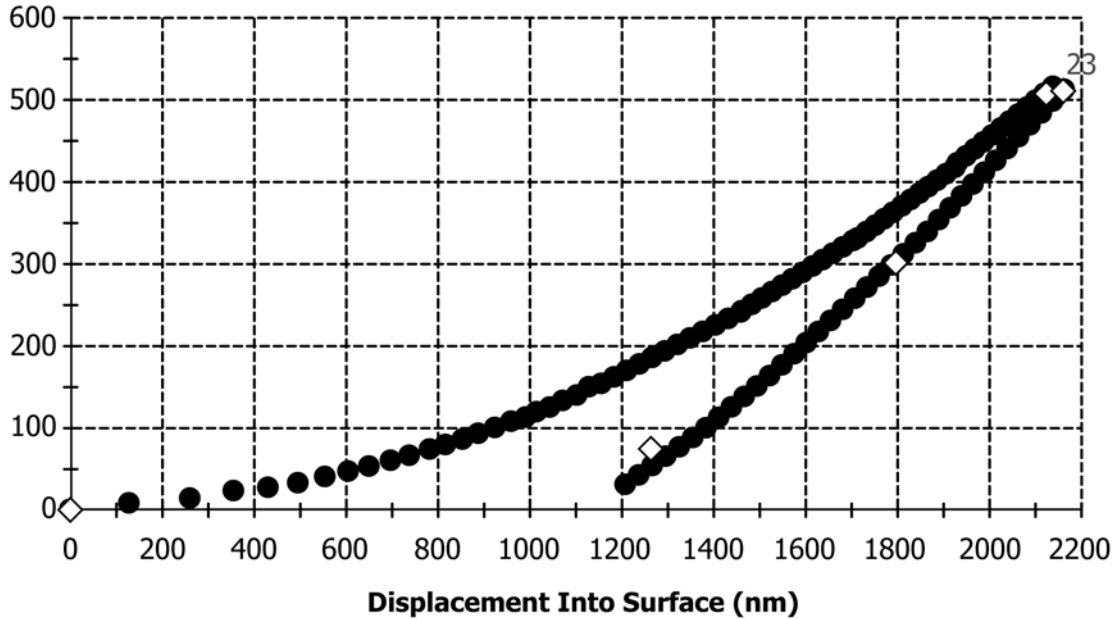$$h_c = 2161.9 \text{ nm} - 0.75*509.9 \text{ mN}/(0.5999 \text{ mN/nm}) = 1524.4 \text{ nm} \quad (X4.9)$$

(4) The nominal area function for a Berkovich indenter is:

$$A_p = 24.5 h_c^2 \quad (X4.10)$$

(5) So, the projected contact area at F_{max} is calculated as:

$$A_p = 24.5*(1524.4 \text{ nm})^2 = 5.693E+7 \text{ nm}^2 \quad (X4.11)$$

Force On Sample (mN)

NOTE 1—Test was performed on fused silica (amorphous quartz) using a modified Berkovich diamond.
FIG. X4.1 Typical Force versus Displacement (*P* versus *h*) Curve

TABLE X4.1 Summary of Intermediate and Final Results

Parameter	Value	Units
P_{max}	509.88	mN
h_{max}	2161.93	nm
B	0.05786	mN/nm^m
h_f	1060.5	nm
m	1.297	–
S	0.5999	mN/nm
h_c	1524.4	nm
A_p	5.69E+07	nm^2
E_r	70.45	GPa
E_{IT}	72.64	GPa
H_{IT}	8.96	GPa

(a) Note—Section A3.7 states that if the indentation depth is less than 6 μm, a refined area function shall be determined for the indenter. For this particular indenter, it was determined in a previous battery of tests that the nominal area function adequately described the diamond shape for indentation depths greater than 1 μm.

(6) The quantity E_r is calculated using Eq X4.2 as:

$$E_r = (\pi/5.693e+7 \text{ nm}^2)^{1/2} * (0.5999 \text{ mN/nm})/2 = 7.045e - 5 \text{ mN/nm}^2$$

$$= 70.45 \text{ GPa} \tag{X4.12}$$

(7) Finally, the indentation modulus, E_{IT}, is calculated from Eq X4.7 using the known elastic properties of the indenter and the Poisson's ratio of the test sample.

$$E_{IT} = (1 - 0.18^2)/(1/70.45 \text{ GPa} - (1 - 0.07^2)/1140 \text{ GPa}) = 72.64 \text{ GPa} \tag{X4.13}$$

(a) Note—An uncertainty range of ±0.1 for a nominal value of Poisson's ratio of 0.25 for the test material produces a ±5 % uncertainty in the calculated value of E_{IT}.

TABLE X4.2 *P* versus *h* Data Used in the Calculation of Results (first 30 of 60 points recorded during unload)

Displacement into Surface, nm	Force on Sample, mN
2161.93	509.88
2151.78	503.19
2140.61	496.53
2129.37	489.81
2117.86	483.12
2106.82	476.40
2095.41	469.71
2083.96	463.04
2072.52	456.31
2060.96	449.64
2049.36	442.96
2037.66	436.23
2026.17	429.54
2014.45	422.87
2002.52	416.15
1990.36	409.46
1978.36	402.74
1966.36	396.07
1955.05	389.38
1943.03	382.66
1931.00	375.97
1919.16	369.28
1906.81	362.57
1894.84	355.88
1882.92	349.20
1870.50	342.47
1859.75	335.78
1848.91	329.09
1836.05	322.40
1810.78	308.98

(8) The indentation hardness, H_{IT}, is calculated from Eq X4.7 as:

$$H_{IT} = 509.9 \text{ mN}/5.693e + 7 \text{ nm}^2 = 8.96e - 6 \text{ mN/nm} = 8.96 \text{ GPa}$$

(X4.14)

X4.9 Report

X4.9.1 The test report shall meet the requirements of Section 9 of this practice.

X4.10 Precision and Bias

X4.10.1 The precision of this test method is based on an interlaboratory study of ASTM E2546, Standard Practice for Instrumented Indentation Testing, conducted in 2014. Participants in six laboratories tested five different materials for both indentation modulus and indentation hardness at two distinct test forces. Every "test result" represents an individual determination, and all participants were asked to report five replicate test results for each material analyzed. All test results are in GPa units. Practice E691 was followed for the study design and analysis of the data; the details are given in ASTM Research Report No. RR:E28-1045.[6]

X4.10.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the

interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

(1) Repeatability limits are listed in Tables X4.3-X4.6.

X4.10.1.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

(1) Reproducibility limits are listed in Tables X4.3-X4.6.

X4.10.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177, except as noted.

X4.10.1.4 Any judgment in accordance with statements X4.10.1.1 and X4.10.1.2 would have an approximate 95% probability of being correct.

X4.10.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

X4.10.3 The precision statement was determined through the statistical examination of 600 test results, from six laboratories, on five materials, at two different test forces.

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E28-1045. Contact ASTM Customer Service at service@astm.org.

TABLE X4.3 Indentation Hardness at 10 mN

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	S_R	r	R
Polycarbonate	0.1862	0.0025	0.0087	0.0070	0.0242
Borosilicate Glass	7.4323	0.2107	0.3134	0.5899	0.8777
Fused Silica	9.4365	0.2090	0.3295	0.5853	0.9227
Silicon 111	12.3263	0.3927	0.4340	1.0995	1.2151
Nano-crystalline nickel	3.6795	0.3364	0.4261	0.9421	1.1931

[A]The average of the laboratories' calculated averages

TABLE X4.4 Indentation Hardness at 500 mN

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	S_R	r	R
Polycarbonate	0.1822	0.0012	0.0181	0.0034	0.0505
Borosilicate Glass	7.2473	0.0336	0.3777	0.0942	1.0577
Fused Silica	9.1157	0.0708	0.3588	0.1983	1.0048
Silicon 111	11.4200	0.2885	0.5770	0.8077	1.6156
Nano-crystalline nickel	3.5333	0.1536	0.2220	0.4301	0.6216

[A]The average of the laboratories' calculated averages

TABLE X4.5 Indentation Modulus at 10 mN

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	S_R	r	R
Polycarbonate	3.0022	0.0514	0.2290	0.1440	0.6411
Borosilicate Glass	63.7120	0.6705	0.9020	1.8774	2.5255
Fused Silica	72.3582	0.7815	0.9486	2.1882	2.6560
Silicon 111	189.7054	4.5226	6.1782	12.6632	17.2990
Nano-crystalline nickel	214.7107	16.6399	18.7378	46.5919	52.4659

[A]The average of the laboratories' calculated averages

TABLE X4.6 Indentation Modulus at 500 mN

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	S_R	r	R
Polycarbonate	2.8588	0.0181	0.1586	0.0508	0.4441
Borosilicate Glass	62.8104	0.3334	0.9492	0.9336	2.6577
Fused Silica	71.5931	0.2377	1.2480	0.6656	3.4944
Silicon 111	191.6166	6.1204	8.0409	17.1370	22.5145
Nano-crystalline nickel	200.7034	3.8885	22.3007	10.8877	62.4418

[A]The average of the laboratories' calculated averages

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<h1 style="text-align:center">SUMMARY OF CHANGES</h1>

Committee E28 has identified the location of selected changes to this standard since the last issue (E2546–07$^{\varepsilon 1}$) that may impact the use of this standard.

(1) X4.10 was revised.

(2) Editorial changes were made throughout.