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.

Designation: E2546 − 15 (Reapproved 2023)

Standard Practice for Instrumented Indentation Testing[1]

This standard is issued under the fixed designation E2546; 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 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *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]

E3 Guide for Preparation of Metallographic Specimens
E74 Practices for Calibration and Verification for Force-Measuring Instruments
E92 Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials
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
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[3]

2.3 *ISO Standards:*

ISO 14577-1, -2, -3, -4 Metallic Materials—Instrumented Indentation Tests for Hardness and Material Properties[4]

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[1] This practice is under the jurisdiction of ASTM Committee E28 on Mechanical Testing and is the direct responsibility of Subcommittee E28.06 on Indentation Hardness Testing.

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

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

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

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ISO 376 Metallic Materials—Calibration of Force-Proving Instruments for the Verification of Uniaxial Testing Machines[4]

3. Terminology

3.1 *Definitions of Terms Specific to 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.

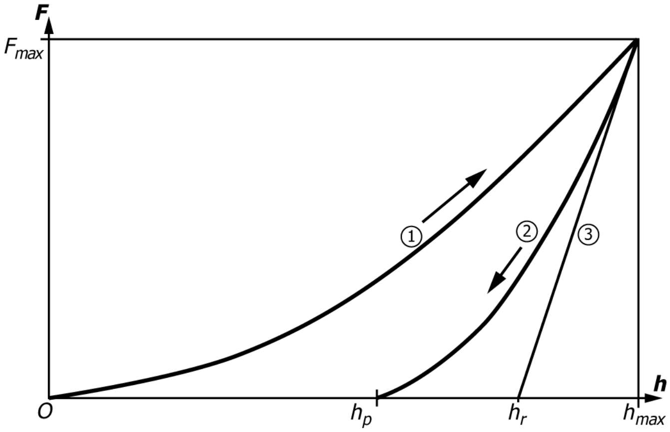
3.1.3.1 *Discussion*—For non-circular impressions, the indentation radius is the radius of the smallest circle capable of enclosing the indentation. The indentation radius is normally used as a guide for spacing of indentations.

3.1.4 *indenter area function [A], 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.



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

FIG. 1 IIT Procedure Shown Schematically

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.

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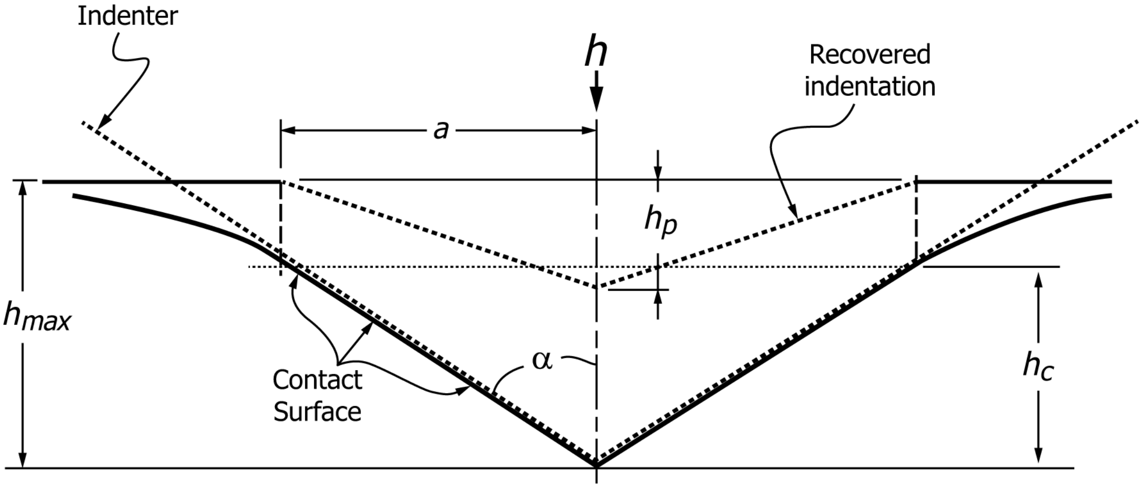
5. Significance and Use

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 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	µm^2
A_p	Projected (cross section) area of indenter at depth h_c	µm^2
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

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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.

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.

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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 °C ± 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.

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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.

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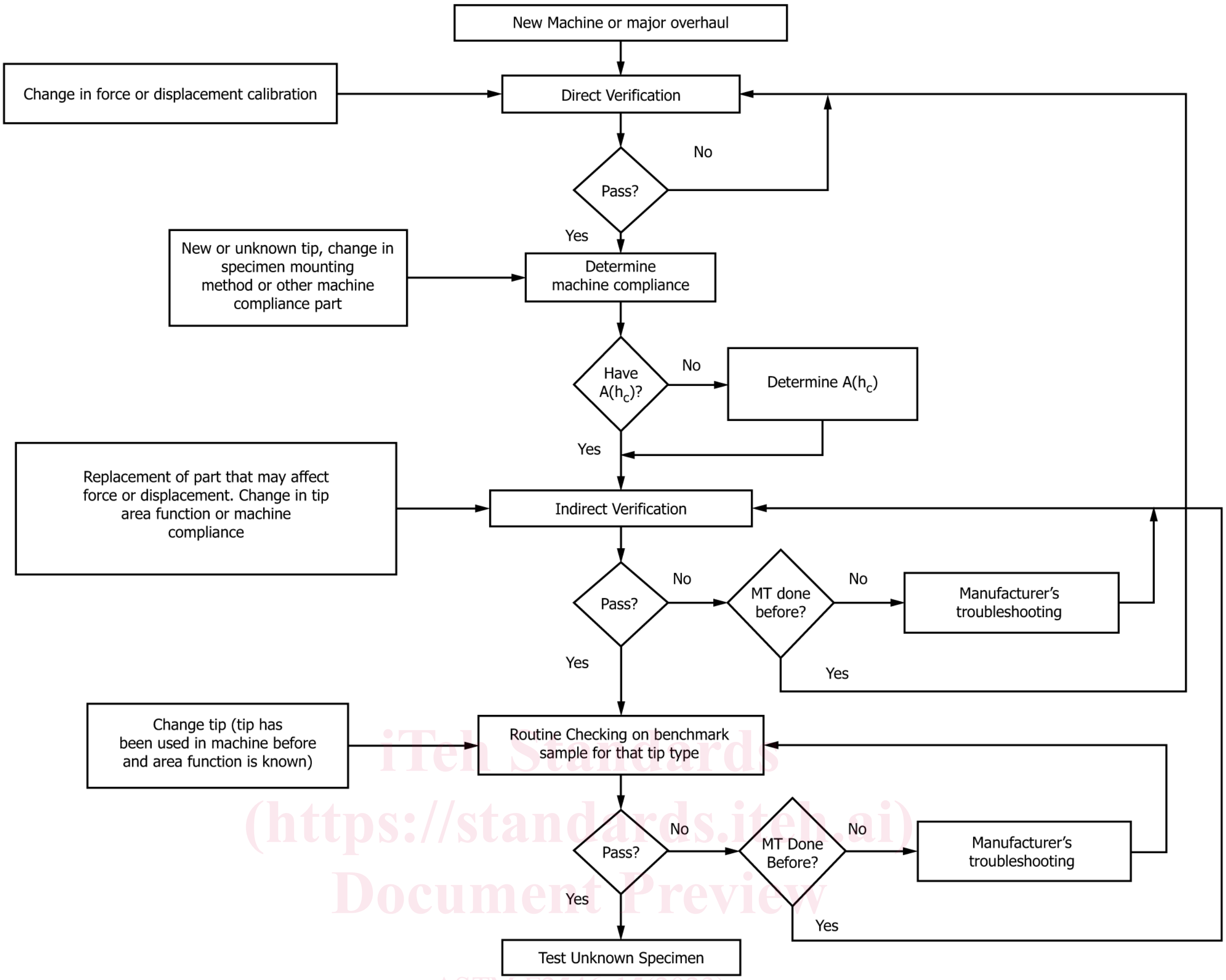

FIG. A1.1 Verification Flowchart

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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.

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.

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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).

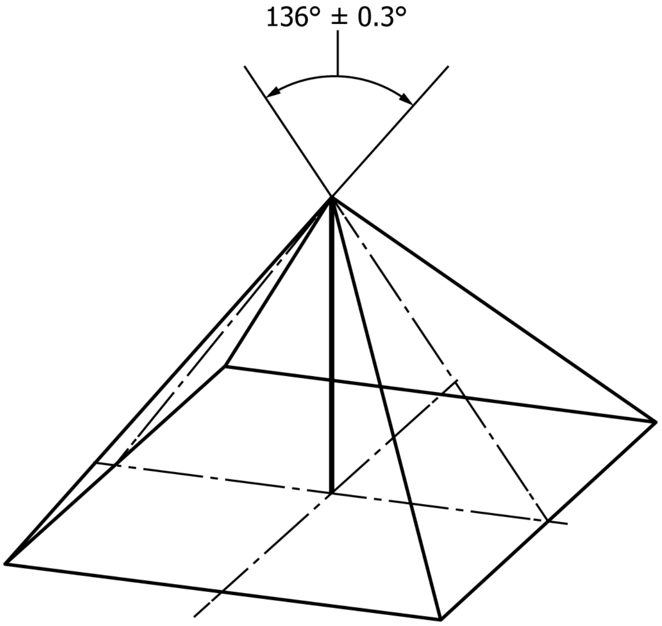

FIG. A3.1 Angle of the Vickers Diamond Pyramid

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

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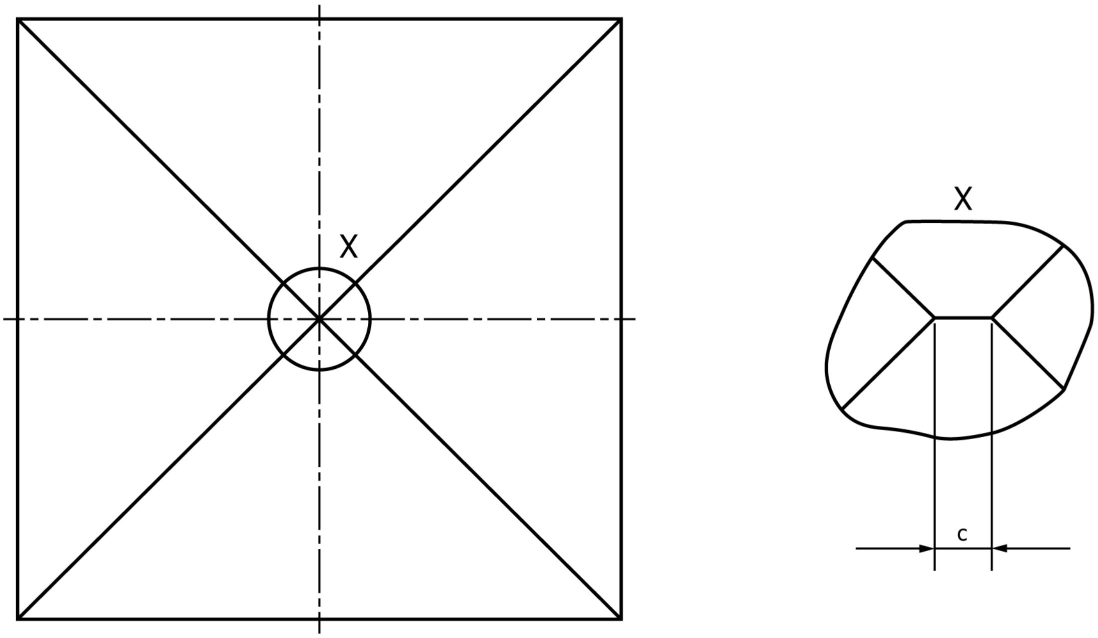

FIG. A3.2 Line of Conjunction at the Tip of the Indenter, Schematically

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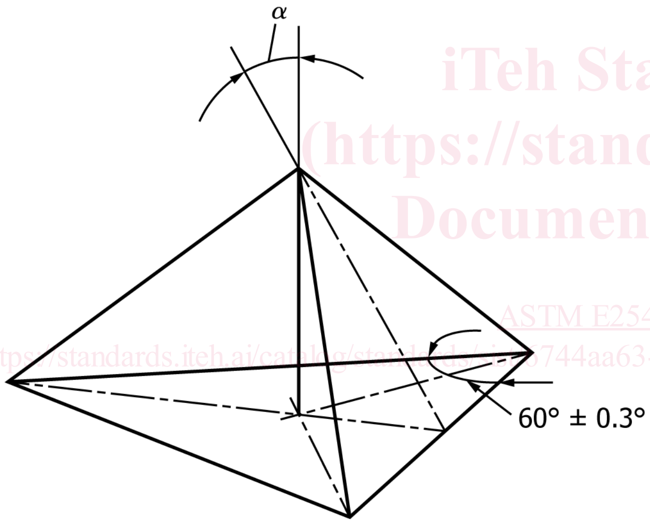

α = 65.03° ± 0.3° for Berkovich indenter
α = 65.27° ± 0.3° for modified Berkovich indenter
α = 35.26° ± 0.3° for corner cube indenters

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}) ≤ 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.)

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