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RECENT CHANGES AND ADDITIONS TO THE HARDNESS TEST METHODS DEVELOPED AND PUBLISHED BY ASTM-INTERNATIONAL

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Abstract – Since the mid 1990s, several factors have influenced the standards development organization ASTM-International to make changes and additions to their published hardness test method standards. These factors included the advent of laboratory quality systems and the resulting need for uncertainty analysis; technological advances in testing equipment and measurement methods; and the recognition that improvement to the methods could be made. The result has been to improve the existing standards and develop new standards to meet evolving needs of industrial users. Some of the more important changes and additions to the ASTM-International hardness standards are discussed.

Keywords ASTM, hardness, standard

1. INTRODUCTION

The conventional hardness test methods, Rockwell, Brinell, Vickers and Knoop, have been used extensively by industry for many decades. The hardness tests have been carried out following prescribed methods specified in hardness test method standards published by national and international standards development organizations such as ASTM-International (ASTM) [1-4] and the International Organization for Standardization (ISO) [5-8]. In recent years, with an increasing number of companies adopting formal quality systems and seeking accreditation, the details and specific requirements of the written standards have come under close scrutiny by the hardness testing and calibration laboratories, as well as, the accreditation auditors. It became evident that some requirements in the ASTM hardness standards were overly strict and in some cases had been ignored by testing and calibration laboratories. Once accredited, the laboratory could no longer ignore any of the requirements specified in the standards. It became important to revise and clarify the standards to make them practical for the industrial users while maintaining the quality of the test results.

It was also evident that some aspects of the hardness test methods had not kept pace with technological advances in hardness testing instruments and components. Additionally, the development of indentation techniques for determining material property information, other than just hardness, prompted the need for standardized methods for instrumented indentation. The ASTM committees having jurisdiction over indentation hardness methods met these needs by revising and improving existing standards and by developing new standards as requested by industry. Some of the more important changes to the hardness test method standards are discussed.

2. CONVENTIONAL HARDNESS METHODS

ASTM has standardized the conventional hardness test methods for many decades. The E 10 Brinell hardness test method standard was first published in 1924, the E 18 Rockwell hardness test method standard in 1932, the E 92 Vickers hardness test method standard in 1952, and the E 384 microindentation (Vickers and Knoop) hardness test method standard in 1969. The E 10 Brinell, E 18 Rockwell and E 92 Vickers hardness standards are under the jurisdiction of ASTM Committee E28 on Mechanical Testing of Metals, while the E 384 microindentation hardness standard is under the jurisdiction of ASTM Committee E04 on Metallography.

Since the mid 1990s, many improvements to these standards have been made, including requirements for indenters, the testing cycle, tester verification procedures, as well as adding new requirements for the accreditation of calibration laboratories, traceability and uncertainty analysis. The changes to the test methods have improved the overall quality of the hardness measurements without significantly affecting the results as compared to historical values.

In this year, 2007, the Rockwell hardness (E 18-07)* and Brinell hardness (E 10-07a)* test methods were approved after having been completely revised and restructured. The structure of the revised standards is to first specify the requirements of the test method followed by mandatory or normative annexes including:

Annex A1: Verification of Testing Machines
Annex A2: Standardizing Machines
Annex A3: Standardization of Indenters
Annex A4: Standardization of Test Blocks.

The annexes are followed by non-mandatory or informative appendices, including discussions of measurement uncertainty.

* Note that the numbers following the standard designation indicate the year of the last revision. The letter "a" at the end of E 10-07a indicates that this is the second revision in 2007.

ASTM began to address the uncertainty of hardness measurements in 2003 by adding an appendix of *Examples of Procedures for Determining Uncertainty* to the E 18 standard. A similar appendix addressing the uncertainty of Brinell hardness measurements was added to E 10-07 in 2007. The appendices provide basic procedures for determining the uncertainty of the following values of hardness:

- *The hardness machine "error" determined as part of an indirect verification*
- *Rockwell hardness value measured by a user*
- *Certified value of a reference test block.*

A significant new addition to E 10-07a and E 18-07 is the requirement that all entities conducting calibrations of reference blocks or indenters, or conducting verifications of the machines used to perform these calibrations must be accredited to the requirements of ISO 17025 by an accrediting body recognized by the International Laboratory Accreditation Cooperation (ILAC) as operating to the requirements of ISO/IEC 17011. The ASTM committee believed that this new requirement for accreditation was needed to ensure that there was some oversight over the quality of these calibrations.

The requirements for what must be reported in test and verification reports and on the reference block calibration certificates of E 10-07a and E 18-07 have been expanded to address newly added requirements of the standards, as well as the requirements of ISO 17025.

The ASTM committee is now considering revising the Vickers hardness standard E 92 to follow the new structure of E 10-07a and E 18-07 and to include some of the improvements of the Brinell and Rockwell standards. The microindentation hardness standard E 384, being under the jurisdiction of a different committee, has been periodically revised in recent years as required, but has maintained its historical format.

3. ASTM E 10 - BRINELL TEST METHOD

3.1 Brinell Indenters

In 2001, ASTM revised E 10-01 to only allow tungsten-carbide ball indenters, eliminating the use of steel balls. In the new E 10-07a version, tungsten-carbide ball indenters of four diameters (10 mm, 5 mm, 2.5 mm, and 1 mm) are specified with no distinction between indenters used for normal-use and calibration machines. The 2 mm diameter ball has been eliminated as a standard ball for this revision.

3.2 Indentation Measuring Devices

An important change to the new E 10-07a is the classification of indentation measuring devices into two types, as either Type A or Type B. The Type A device includes microscopes having movable measuring lines with some type of indicator or computerized measuring system, or an image analysis system. The Type B device is a hand-held microscope with fixed measuring lines, usually having a 20× or a 40× magnification. The Type B measuring device is limited to measuring indentation diameters made by 5 mm or 10 mm ball tests. It had become apparent that Type B devices had difficulty in meeting the accuracy requirements specified in previous versions of E 10. The coarse graduation lines of Type B devices make it difficult to determine an accurate quantitative value for the measurement error. Although Type B devices are not ideal measuring instruments from a standardization point of view, they are in common use in industry and should continue to be allowed. Consequently, the two types of measuring devices are now specified differently.

Type A devices are specified with tolerances on the minimum indicator resolution, and are verified for accuracy by determining the measurement error with respect to a reference length standard, such as a graduated-line glass stage micrometer. Type B devices have tolerances on the maximum spacing of the graduated measurement lines, and, in keeping with industrial practice, verification is accomplished by positioning the measuring device such that the lines of the device line up with the lines of the stage micrometer as closely as possible. If any lines of the measuring device do not, at least partially, overlap the corresponding lines of the stage micrometer, then the measuring device must be adjusted. Because of the differences in the measurement accuracies and verification procedures for Type A and Type B devices, the type of measuring device that is used must be indicated on the test report.

3.3 Brinell Testing Machine Verifications

Direct verification independently verifies whether the major components of a hardness machine are within acceptable tolerances. Indirect verification assesses the measurement performance of the testing machine by making hardness measurements on reference blocks and comparing the measurement values with the certified values of the blocks. Tolerances are specified for the allowed error between the verification measurements and the certified block values.

In previous versions of E 10, verification of in-service Brinell hardness testing machines could be performed either by direct verification or by indirect verification. Both methods were equally acceptable. With the new E 10-07a, it is now mandatory to conduct periodic indirect verifications at least every 18 months, while direct verification is required only when a machine is new, or when adjustments, modifications or repairs are made, or when a testing machine fails an indirect verification.

The direct verification of a Brinell testing machine verifies whether the applied test forces, the indentation measuring system, the loading rate and the dwell time at total force are within specified tolerances. Direct verifications of the loading rate and the dwell time at total force, referred to as the testing cycle, are new requirements to be verified by the testing machine manufacturer at the time of manufacture, or when a problem with the testing cycle is suspected. Verification of the testing cycle is not required as part of the direct verification at other times.

As with previous versions of E 10, the indirect verification process is designed to verify each test force and ball size to be used. The new E 10-07a states the requirements more clearly than in previous versions. The new standard also recommends that, before performing any cleaning, maintenance, or verifications of the testing machine, the as-found condition and performance of the testing machine be evaluated, and provides a suggested as-found evaluation procedure.

A new verification requirement of E 10-07a is that prior to conducting indirect verification hardness tests, the measuring device must be verified by measuring the diameters of the smallest and the largest reference indentations in the reference blocks that will be used for the indirect verification. Tolerances are specified for how closely the diameter measurements must agree with the certified diameter values, which vary depending on whether the measuring device is Type A or Type B. This procedure is also recommended as a trouble-shooting tool in the case that the testing machine fails a daily verification. As an alternative to measuring reference indentations, the measuring device may undergo direct verification.

3.4 Calibration of Reference Blocks

The new revision of E 10-07a defines a higher level of Brinell hardness machine designated as a “standardizing machine,” which expands the requirements set out in previous versions of E 10 for the machine used to calibrate reference blocks. The standardizing machine now requires the use of a Type A indentation measuring device, tighter controls on the laboratory environment (i.e., temperature and humidity control) and expanded verification requirements. All calibrations of reference blocks must be made using a standardizing machine.

In previous versions of E 10, the standardizing machine was required to meet the same verification requirements as the normal-use Brinell testing machine discussed previously. The new E 10-07a now requires the standardizing machine to undergo direct verification at least 12 months prior to reference block standardizations. A traditional periodic indirect verification of the standardizing machine is no longer required; however, a procedure is specified for monitoring the standardizing machine by performing monitoring verifications each day that test block calibrations are made.

Monitoring verifications are to be conducted prior to the test block calibrations, and may be made either by direct verification or by performance verification using reference blocks. In the case that monitoring verifications are to be made by performance testing, Brinell hardness tests are to be performed on at least one mid-range reference block for each force level and for each ball size that will be used on that day.

As discussed previously, reference indentations are now required to be measured as part of the indirect and daily verifications of a Brinell hardness machine. A new requirement of E 10-07a is that in addition to calibrating a reference block for the average hardness of the test surface, one or more of the calibration indentations must be certified for the measurement of the diameter and will be referred to as reference indentations.

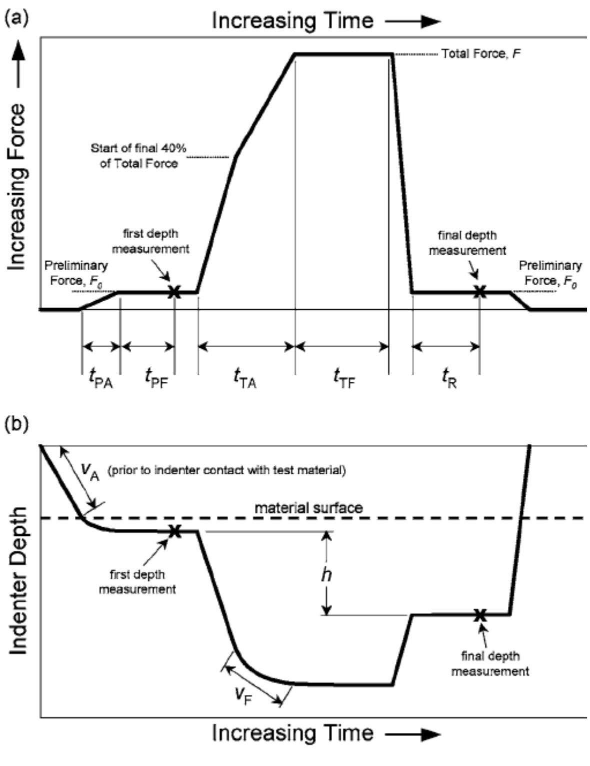

Fig. 1. Schematic of Force vs Time (a) and Indenter Depth vs Time (b) of a Rockwell test illustrating the test cycle parts

4. ASTM E 18 - ROCKWELL TEST METHOD

4.1 Rockwell Testing Cycle

The Rockwell hardness test requires two force levels to be applied to the indenter at specific loading rates, and the forces are to be held constant for specific time durations. The manner in which this is done is referred to as the testing cycle of the Rockwell hardness test (see Fig. 1). It is well known that varying different parts of the testing cycle produces different measurement results [9]. To account for this, the parameters of the testing cycle that have the largest effect on the hardness result are specified with tolerances in the test method standards. In industry, it is usually preferred to run tests as fast as possible in order to conduct the largest number of tests in the shortest time. In the case of Rockwell hardness testing, a fast test time is accomplished by using fast loading rates and short constant load dwell times. Unfortunately, an incremental change in a dwell time will produce the largest variation in the measurement results when short dwell times are used.

Recognizing these problems, ASTM revised the tolerances for the E 18-07 revision to reduce measurement differences, but without setting the requirements outside the capabilities of any hardness machines currently in service. The testing cycle requirements were revised to prevent very short dwell times while better aligning the tolerances with the newly defined Rockwell C scale testing cycle proposed by the Working Group on Hardness of the Consultative Committee on Mass and Related Quantities of the International Committee of Weights and Measures (BIPM) [10]. The previous and the newly defined ASTM testing cycle and the WGH Rockwell C (HRC) testing cycle are given in Table 1.

TABLE 1. Parameter values of the previous and newly defined ASTM testing cycles and the WGH Rockwell C (HRC) testing cycle

Test Cycle Parameter	ASTM E 18-05	New ASTM E 18-07 (Testing Machine)	New ASTM E 18-07 (Calibration Machine)	CCM-WGH HRC Reference Value
Indenter contact velocity, v_A	Without shock or vibration	≤ 2.5 mm/s (recommended)	≤ 1.0 mm/s	Not defined
Dwell time for preliminary force, t_{PF}	≤ 3.0 s	0.1 to 4.0 s (when the time to apply the preliminary force t_{PA} ≥ 1 s, then calculate this parameter as $t_{PA}/2 + t_{PF}$)	2.0 to 4.0 s (when the time to apply the preliminary force t_{PA} ≥ 1 s, then calculate this parameter as $t_{PA}/2 + t_{PF}$)	3.0 s (calculate this parameter as $t_{PA}/2 + t_{PF}$)
Application time for additional force, t_{TA}	1.0 to 8.0 s	1.0 to 8.0 s	1.0 to 8.0 s (Velocity criteria are recommended)	Not defined; (Velocity criteria are required.)
Dwell time for total force, t_{TF}	≤ 3.0 s	2.0 to 6.0 s	4.0 to 6.0 s	5.0 s
Dwell time for elastic recovery, t_R	Not defined	0.2 to 5.0 s	3.0 to 5.0 s	4.0 s

4.2 Rockwell Indenters

Perhaps the most significant changes to the new E 18-07 have been to the requirements for the diamond and ball indenters. All Rockwell indenters must now be serialized, including the ball holders of ball indenters. When it is not practical to mark the serial number on the indenter due to size limitations, the serial number may be marked on the indenter container.

Indenters are required to be directly verified for the correct geometric features, and indirectly verified for performance by direct performance comparison with a higher level indenter in the case of diamond indenters, and by testing reference blocks in the case of ball indenters. The geometry is verified when the indenter is new, often by the manufacturer. Performance is also verified when the indenter is new or when suspected damage has occurred. The only exception is for the Class A diamond indenter, used for reference block calibrations, which must be verified for performance within 12 months prior to block calibrations.

Diamond indenters: In prior versions of E 18, two levels of diamond indenters were specified: the indenter for normal testing and the indenter for calibrating test blocks, the calibrating indenter having tighter tolerances than a normal-use indenter for performance and geometric features of the diamond. The diamond indenter was verified for performance by testing a minimum of two reference blocks of different hardness levels.

The new E 18-07 now specifies three levels of diamond indenters designated as Class B, Class A and Reference indenters. Class B indenters are intended for every day use with Rockwell hardness testing machines. Class A indenters are intended for the standardization of Class B indenters and for the standardization of test blocks. Reference indenters are intended for the standardization of Class A indenters, and their performance is verified with a National Metrology Institute. Each class of indenter is verified for performance by comparison with a higher class indenter with tighter acceptability tolerances for each higher class.

Each class of diamond indenter is also verified for correct geometry. Previous versions of E 18 specified that indenters used for calibrations (now Class A) meet the following geometrical requirements: included angle of 120 ± 0.1°; mean radius of 0.200 ± 0.005 mm; and the radius in each measured section of 0.200 ± 0.007 mm. At the time the new E 18-07 was developed, the ASTM committee believed that diamond indenters meeting these tolerances were not reliably available on the world market. Consequently, the tolerances for the geometric features of the Class A and Reference diamond indenters have been temporarily widened to the levels of Class B indenters until such time as indenters having tighter tolerances become reliably available (see Table 2).

Class B and Class A diamond indenters may be certified either for use only with the regular Rockwell scales (i.e., HRA, HRC and HRD), or for use only with the superficial scales (i.e., HR15N, HR30N and HR45N), or for use with all Rockwell diamond indenter scales. Although this was common practice in the past, previous versions of E 18 did not clearly address this.

Ball Indenters: The new E 18-07 now specifies two levels of ball indenters designated as Class B and Class A indenters. Class B indenters are intended for every day use with Rockwell hardness testing machines. Class A indenters are intended for the standardization of test blocks. In the new E 18-07 version, as well as in prior versions, ball indenters of four diameters, 1.588 mm ($^1/_{16}$ in.) , 3.175 mm ($^1/_8$ in.), 6.350 mm ($^1/_4$ in.) and 12.70 mm ($^1/_2$ in.), are allowed with the distinction between normal-use (Class B) and calibration indenters (Class A) being a tighter tolerance on the ball diameter for the calibration indenter. However, prior versions of E 18 allowed either steel or tungsten-carbide balls to be used, and did not require a separate verification of the indenter's performance.

In the 1990s, many ATSM committee members observed that a major cause of measurement error in industrial Rockwell hardness testing was steel indenter balls that had become damaged from overuse, accidental impact of the test piece, or testing too hard a material. In 2002, a revision to E 18-02 was approved to allow the use of tungsten carbide ball indenters with the intent to eventually eliminate the use of steel ball indenters. A problem with changing to the tungsten carbide indenter is that it produces a slightly lower hardness value than when using a steel ball indenter. Under load, the two types of balls elastically deform differently resulting in slightly different indentation behavior producing different hardness values. The ASTM committee decided that the benefit of reducing indenter ball damage through the

Table 2. Geometrical requirements for a calibration grade (Class A) diamond indenter as specified in previous and the new versions of E 18.

Parameter	Previous E 18	New E 18-07
included angle	120 ± 0.1°	120 ± 0.35°
mean radius	0.200 ± 0.005 mm	0.200 ± 0.010 mm
radius in each measured section	0.200 ± 0.007 mm	0.200 ± 0.015 mm
local deviations from a true radius	≤ 0.002 mm	≤ 0.002 mm

use of the harder carbide ball far outweighed any problem associated with the small shift in the hardness result.

The new E 18-07 now specifies that tungsten carbide balls must be used for all testing, except in the very narrow case of testing thin sheet tin mill products specified in ASTM Specifications A 623 and A 623M using the HR15T and HR30T scales with a diamond spot anvil. This exception continues to be allowed due to the large measurement differences between using a steel or tungsten carbide ball to test this thin sheet material, and the need for uniformity with historical steel-ball test data.

Ball indenters commonly consist of a holder, a cap, and a ball that can be replaced. The new E 18-07 now allows one-piece fixed-ball indenters provided the indenter meets the same requirements as removable ball indenters. Fixed ball indenters were not allowed by some previous versions of E 18.

Other new requirements for the ball indenter have been added. The ball indenter is now required to pass performance tests on a minimum of one reference block. Additionally, the protrusion of the ball outside the ball holder is now specified and must be verified, either by direct measurement or by performance tests on a soft reference block ≤ 10 HRBW. These verifications are required when the indenter is new and are usually performed by the manufacturer.

4.3 Rockwell Testing Machine Verifications

In previous versions of E 18, the specified requirements for verifying Rockwell hardness testing machines were not well specified and often led to varying procedures being performed by different calibration agencies. The new E 18-07 more clearly states the requirements for the *direct*, *indirect* and *daily* verifications of the testing machine and the schedule for when the verifications must be performed.

A direct verification of a Rockwell hardness machine determines whether the application of the test forces, the depth-measuring system, machine hysteresis and the testing cycle are within specified tolerances. As in previous editions of E 18, direct verification is required only when a testing machine is new, moved, or when adjustments, modifications or repairs are made, or when the machine fails an indirect verification.

Verification of the hysteresis and the testing cycle, as part of a direct verification, are new requirements for E 18-07. The testing cycle is to be verified by the testing machine manufacturer at the time of manufacture, and when the testing machine is returned to the manufacturer for repair, or when a problem with the testing cycle is suspected. Verification of the testing cycle is not required as part of the direct verification at other times.

The new E 18-07 requires that the testing machine must undergo periodic indirect verifications of each Rockwell scale that will be used prior to the next indirect verification. Hardness tests made using Rockwell scales that have not been verified within the schedule do not meet the standard. This requirement was not clearly stated in previous versions of E 18. As with the new Brinell standard E 10-7a, the new E 18-07 also recommends that the as-found condition of the testing machine be evaluated prior to performing direct and indirect verifications, and provides a suggested as-found verification procedure.

The new E 18-07 also clarifies that the indirect verification is only valid when the testing machine is used with the specific user's indenters that were used for the indirect verification. Before any other indenter may be used for testing, it must be verified with the testing machine. A procedure to verify a new indenter has been added to E 18-07, which requires performing a subset of a full indirect verification. In this case, the verification may be performed by the user. This requirement does not apply to simply changing an indenter ball.

A daily-verification to monitor the performance of the testing machine is now required each day that hardness tests are to be made, and is recommended whenever the indenter, anvil, or test force is changed. This was only a recommended verification check in previous versions of E 18.

4.4 Calibration of Reference Blocks and Indenters

The new revision of E 18-07 defines a higher level of Rockwell hardness machine designated as a "standardizing machine," which expands the requirements set out in previous versions of E 18 for the machine used to calibrate reference blocks, but is now also applicable for the machine used for the performance verifications of indenters. The standardizing machine has tighter tolerances than a normal-use testing machine for various components and functions of the machine, including the applied forces, the testing cycle to be used (see Table 1), a higher class of indenters (i.e., Class A or higher), and the laboratory environment (i.e., temperature and humidity control). All calibrations of reference blocks and indenters must be made using a standardizing machine.

A standardizing machine is now required to undergo direct verification at least every 12 months. Periodic direct verification is a new requirement starting with this edition of the standard. In previous editions of E 18, direct verification was required only when a standardizing machine was new, moved, or when adjustments, modifications or repairs were made.

The new E 18-07 specifies indirect verification differently for a standardizing machine than for a regular testing machine. Ultimately, it is required that an indirect verification of all Rockwell scales to be used for standardization testing must be made within 12 months prior to standardization. However, immediately following a direct verification, a limited number of scales must undergo indirect verification. The scales are chosen to test the performance of the standardizing machine at each force level that the standardizing machine is capable of applying. This procedure was developed because a reference block calibration laboratory often does not standardize blocks for all 30 Rockwell hardness scales within a 12 month period. It is not reasonable to require the laboratory to spend the excessive amount of time needed to verify all 30 Rockwell scales when not all scales may be used. The limited number of verifications will ensure that the machine is operating correctly at each force level at the time of the direct verification, and then each scale to be used will eventually undergo indirect verification prior to the block standardization.

The daily verification requirements for a standardizing machine are also handled differently by the new E 18-07. The verification is more extensive than the daily verification of a normal-use testing machine. A procedure was devised to test at least two "monitoring" test blocks in the appropriate hardness ranges that bracket the hardness level to be standardized. The monitoring tests are to be made before and after each lot is standardized, and at the end of each day and the start of the following day when a single lot is standardized over multiple days. A procedure for calibrating monitoring blocks is provided.

5. VICKERS AND KNOOP TEST METHODS

ASTM currently specifies the Vickers hardness test in two different standards. ASTM E 92 specifies what is sometimes referred to as "heavy-load Vickers", that is, tests using applied forces of 9.807 N (1 kgf)[†] to 1.177 kN (120 kgf)[†]. ASTM E 384 specifies microindentation Vickers and Knoop hardness for forces of 9.807 N (1 kgf)[†] and below. As discussed previously, the two standards are under the jurisdiction of different committees within ASTM.

At this time, E 92 is being revised to adopt the structure of the new E 10-07a and E 18-07 standards. The ASTM committee is also proposing to expand the lower applied force limit down to 1.961 N (0.2 kgf)[†]. This proposed change was prompted because there are many Vickers testing machines that are capable of applying forces spanning portions of the allowed ranges of both E 384 and E 92. Agencies that perform verifications of these machines must now perform two separate verifications to meet the two different standards. This proposed change will help eliminate this complication for the E 92 type of testing machines.

The E 384 standard will continue to be applicable to the Vickers and Knoop microindentation type of testing machines. A significant change under consideration for E 384 is to reduce the number of calibration measurements required to calibrate a reference block from 25 (five groups of five) to only five indentations. Studies of historical records of Vickers and Knoop microindentation reference block calibrations over recent years have shown that five indentations provide essentially the same certified value and data spread as 25 indentations. The greater number of measurements had been needed in the past in order to pass material uniformity requirements. With improvements to the block material and standardizing machine repeatability, the added indentations are believed to no longer be necessary.

6. INSTRUMENTED INDENTATION METHODS

The newest and fastest growing activity in the field of indentation and hardness standardization is Instrumented Indentation Testing (IIT). The advantage of the IIT test is that it can be used not only for the determination of indentation hardness, but other material property characteristics as well. Standardization of the test method has now become an important issue for industry. ASTM committee E28 has addressed the needs of industry by developing a new E 2546-07 Standard Practice for Instrumented Indentation Testing, which is in the final approval stage. The Standard Practice establishes the requirements, accuracies, and capabilities needed by an instrument to successfully perform the test and produce the IIT data. It does not define the analyses necessary to determine material properties. Those analyses are left for other test methods.

[†] Kilogram-force (kgf) units are included because of historical use by ASTM.

7. SUMMARY

The conventional hardness and indentation test methods of ASTM-International have been significantly revised in recent years to improve the tests, clarify requirements and to meet the needs of the users of the standards. To address standardization issues related to the evolving measurement field of instrumented indentation testing, a new standard has been developed. The development and improvement of ASTM indentation hardness standards is an ongoing and active process to meet the needs of the industries that perform these measurements.

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