Developing definitions of conventional hardness tests for use by National Metrology Institutes

ARTICLE INFO

ABSTRACT

This paper describes the process used by the Consultative Committee for Mass and Related Quantities - Working Group on Hardness (CCM-WGH) of the International Committee of Weights and Measures (CIPM) to develop international definitions of the conventional Rockwell, Brinell, Vickers and Knoop hardness test methods, for use by the National Metrology Institutes (NMI) that standardize hardness measurement.

1. Introduction

The conventional hardness test methods, Rockwell, Brinell, Vickers and Knoop, are ordinal quantity measurements that are dependent on a defined test method. As a result, a variation in any one test parameter usually leads to a different hardness measurement result.

In today's international commerce, conventional hardness testing is almost exclusively conducted in compliance with the Rockwell, Brinell, Vickers and Knoop hardness test methods specified by either ASTM-International (ASTM) [1] or the International Organization for Standardization (ISO) [2]. These test methods specify requirements for the significant parameters of the hardness tests.

The ASTM and ISO hardness standards were developed as test methods for use in many different industrial testing applications. Consequently, the test parameter values are usually specified to be within ranges of values to accommodate the different testing needs and designs of hardness testing machines. Adhering to the requirements of the test method standards provides an acceptable level of confidence that the measurement result will be appropriate for its intended need. As long as the parameters of their hardness machines differ within the allowances of the test method, there will be acceptable agreement between the measurements of a supplier and customer. While this potential level of measurement bias is acceptable for industrial testing, better agreement is needed between countries at the highest national level to ensure international equivalence for global testing.

International measurement equivalence between National Metrology Institutes (NMI) is the role of the International Committee of Weights and Measures (CIPM). Within the CIPM is the Consultative Committee for Mass and Related Quantities (CCM) [3], and within the CCM is the Working Group on Hardness (CCM-WGH). The CCM-WGH members are the world's NMIs that standardize hardness measurement for their country and transfer hardness reference values for traceability purposes. One goal of the CCM-WGH is to develop more specific international definitions of the hardness tests for use by NMIs to reduce the measurement differences between countries.

The overall traceability framework is for the industries of different regions of the world to comply with the consensus international test definitions and achieve traceability to the national hardness standards of NMIs through a chain of measurement comparisons. The equivalence between NMIs is determined through international comparisons planned and executed by the CCM-WGH.

2. Conventional hardness tests

The conventional hardness test methods, Rockwell, Brinell, Vickers and Knoop, follow a similar basic test procedure. An indenter of known shape and material is forced into a sample under a known force. The hardness value is based on a specific measurement of the resulting indentation. The Rockwell hardness test is a one-step process where the depth of indentation is measured during the indentation process to provide the hardness value. The Brinell, Vickers and Knoop hardness tests follow a two-step process where the sample is first indented, then a specific geometrical characteristic of the indentation is measured to determine the hardness result.

2.1. Rockwell hardness

Fig. 1 illustrates a Rockwell hardness test cycle, where the applied force during the test cycle is shown at the top portion of the figure, and the corresponding indentation behavior is shown below. The Rockwell hardness test applies two levels of force to the indenter with periods of the force being held constant (dwell times) and the indentation depth is measured twice. The two measurements of the indentation depth are indicated by red arrows. The Rockwell hardness value is calculated based on the difference between the initial and final depth measurements Δd (mm) (see Fig. 1).

There are 30 Rockwell scales defined by ASTM (ISO defines 15 of these), each employing different applied forces and/or type of indenter. The Rockwell scales best suited for testing harder materials utilize a conically shaped diamond indenter with a spherical tip, and the Rockwell scales for testing softer materials use tungsten carbide composite ball indenters of four different diameters.
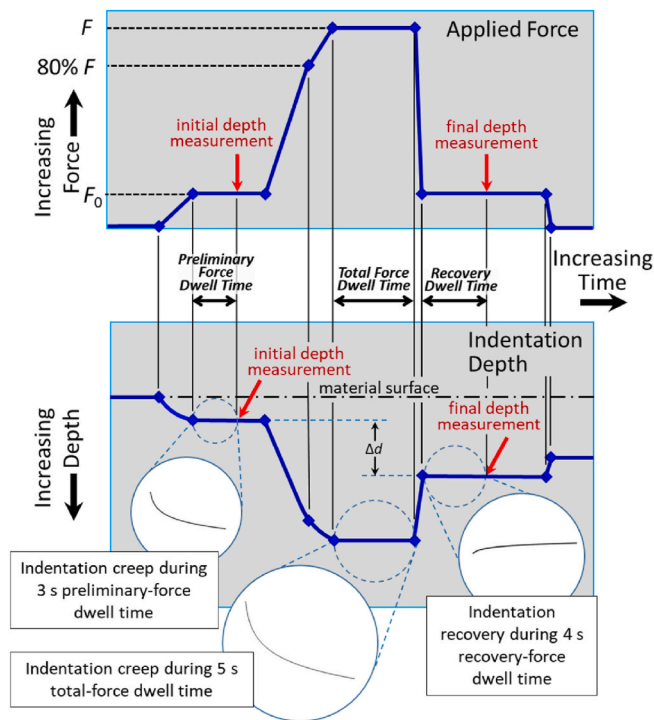
Fig. 1. Schematic of the Rockwell hardness test illustrating the force application and corresponding indentation depth during the test cycle. Expanded views are displayed of the material creep and material recovery occurring during force dwell times.

2.2. Brinell, Vickers and Knoop hardness

Brinell hardness is measured by indenting a material normal to its surface with a tungsten carbide composite ball indenter. The specified forces can range from 9.807 N (1 kgf) to 29.42 kN (3000 kgf). ASTM and ISO define many Brinell scales, each designated by the combination of the applied force and the indenter ball diameter. The Brinell hardness value is calculated based on the applied force divided by the resulting curved surface area of the indentation.

Since measuring the curved surface area of the indentation would be time consuming and extremely difficult, the Brinell test method specifies that the surface area be estimated by measuring the projected area of the indentation at the sample surface. Historically, the projected area of the indentation has been determined from the mean diameter, d_B, of two

Fig. 2. Surface view of the Brinell, Vickers and Knoop indentations illustrating the dimensions that are measured.

perpendicular optical measurements of the projected indentation diameter (d_{B1} and d_{B2}) as shown in Fig. 2. This technique continues to be used today as well as automatic video systems such as those that fit a circle to the entire observed projected area of the indentation.

The Vickers and Knoop hardness tests follow the same basic procedure as the Brinell test. Both tests apply a specified force to indent a material normal to its surface, followed by the measurement of the resulting indentation. The Vickers test uses a square-based diamond pyramidal indenter of specified dimensions. Vickers hardness is based on the indentation test force divided by the surface area of the resulting indentation. The indentation surface area is calculated from the mean diagonal length, d_V, of the two diagonal lengths (d_{V1} and d_{V2}), which are optically measured at the indentation surface as shown in Fig. 2. The Knoop test procedure is the same as for the Vickers test but uses a rhombic-based, pyramidal diamond indenter. The Knoop hardness number is based on the indentation test force divided by the projected area of the indentation. The projected area of the indentation is calculated from the optical measurement of the length of the long diagonal, d_K, as shown in Fig. 2.

3. Developing definitions

The CCM-WGH process for developing hardness test definitions typically begins by evaluating the test parameter values being used by NMIs to standardize their national hardness scales, as well as the values specified by the consensus test method standards. For some test parameter values, such as the applied forces, indenter geometries and testing temperature, the choice of test parameter value is clearly defined by the consensus test method standards and adhered to by NMIs. However, for some test cycle parameters, the most appropriate value is not clear and for other parameters, such as measurement of the indentations, improved definitions may be required.

Before defining values for the various test parameters, the CCM-WGH must understand how each parameter affects the hardness result by determining sensitivity coefficients for the proposed values. This is important for two reasons: to confirm that the sensitivity coefficients are sufficiently small that the CCM-WGH can be confident that the parameter is appropriate for an international definition, and for calculating measurement uncertainties due to deviations from the defined parameter values. For these ordinal hardness quantity measurements, determining sensitivity coefficients is primarily accomplished empirically by conducting hardness tests that vary the parameter values while holding all other parameters constant. For some parameters, such as indenter ball material, modelling programs have been useful. Many studies to determine sensitivity coefficients of hardness test parameters have been made by NMIs and the data is collected by the National Physical Laboratory (NPL) in the UK for open access on their website [4].

The ideal goal of the CCM-WGH is to define each test parameter as a single value and not ranges of values as is done by consensus test method standards. However, for several test cycle parameters, this is not realistic. For example, a force application time or indentation velocity may be ideal for one material or hardness level but inappropriate for another. Since it would be impractical to define a different hardness test parameter for each material or hardness level, an appropriate range is defined, usually narrower than specified by the consensus test method standards.

4. Definition parameters

For each of the conventional hardness tests, the parameters that influence the hardness result, and thus must be defined, fall into two main categories: the hardness machine components and the testing cycle parameters. Discussion of these parameters follows in this section with examples of specific technical issues that were debated within the CCM-WGH and the solutions that were agreed to. Also discussed are issues for which consensus solutions have not yet been realized.
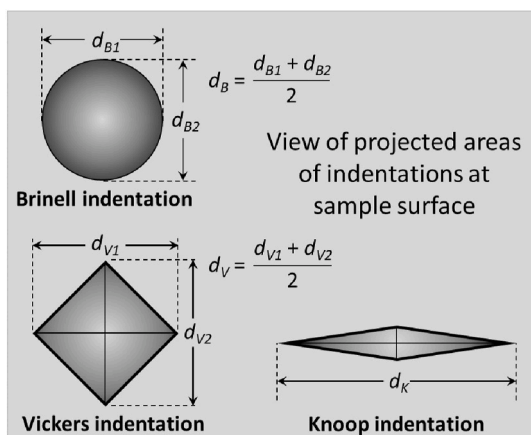
4.1. Hardness machine components

From the brief descriptions of the conventional hardness tests given in Section 2, several parameters of the functions of the hardness machine immediately stand out as needing to be defined: the test forces, the indenter, and the measurement of the indentation.

The test forces used for the conventional hardness tests are inherent to the principles of the methods and are generally well defined although there was a recent debate on the values to define. The hardness test methods originally defined test forces as whole numbers in kgf units (e. g., 10 kgf). Later, the consensus hardness standards specified the test forces in N units by multiplying the kgf values by the constant 9.806 65. and then rounding the values. The CCM-WGH debated whether the test force values should be defined to include all the digits of the conversion, or as the rounded values specified in the test method standards. The CCM-WGH chose to use this conversion from kgf as the defined N force values after it was pointed out that the forces of many primary NMI hardness machines (applied by weights in many cases) were calibrated to the exact N values converted from the kgf values multiplied by 9.806 65.

The test method standards clearly define the ideal indenter geometries, which the CCM-WGH has also adopted for their definitions. However, the CCM-WGH found that the measurement of some aspects of the geometry needed to be addressed to improve measurement agreement. For the Rockwell diamond indenter, the ideal shape is conical with a 120° included angle and a spherical tip having a 0.2 mm radius blending tangentially with the cone. Any deviation in either the cone angle or tip radius will move the blend point of these two parameters. If these two geometric parameters are measured from the location of the ideal blend point as originally implied by the test method standards, then the measurement of either the cone angle or tip radius can include area of the other parameter [5]. This has caused indenters to exceed geometrical limits and fail acceptance. With this understanding, the CCM-WGH has defined the Rockwell diamond indenter to have the ideal dimensions with a recommendation that the blend area not be included in the measurements of the cone angle or tip radius. This information has also made its way into the test method standards.

In the cases of the Vickers and Knoop diamond indenters, the ideal geometry is well defined in the test method standards. In practice, however, a perfect tip geometry is never achieved by diamond machining capabilities, and the tip dimensions needing to be measured become smaller than is possible to resolve with optical measuring systems. Whether or not it will be necessary to stipulate specific measurement practices or adopt new acceptance criteria will have to be addressed when the CCM-WGH moves to define these hardness tests.

For the Rockwell and Brinell ball indenters, it isn't the indenter geometry that is the challenging issue, but rather the material of the ball that needs to be defined. Rockwell and Brinell indenter balls are a tungsten carbide composite specified by the test method standards for material hardness, density and chemistry, with limit ranges on these parameters. The issue is how the ball elastically deforms, or changes shape, under the pressure of indentation. The change in ball shape during indentation alters the indentation stresses and can significantly affect the hardness result. Whether to specifically define the chemistry of the ball material, or to define a material property of the ball material, such as Young's modulus, is still being debated by the CCM-WGH. An entirely different solution has been suggested that the CCM-WGH maintain a large stockpile of Rockwell and Brinell indenter balls which would become the CCM-WGH ball definition for NMI use.

For the Brinell, Vickers and Knoop hardness tests, the measurement of the indentation is recognized as the major contributor of error and uncertainty in the hardness result. As discussed in Section 2, the hardness values are calculated from the measurement of the resulting indentation. Measurement agreement between NMIs can only occur when their respective measurement systems measure the same physical features of the indentation. There are two issues that hinder this. The

Brinell indentation edge and the diagonal tips of the Vickers and Knoop indentations are difficult to define and can be open to interpretation. Equally problematic is that two different indentation measuring systems utilizing optical microscopes can perceive the same Brinell edge or diagonal tip differently.

In the case of a Brinell indentation, the challenge is to determine the edge of the indentation, which is curved due to the pileup or sinking-in of the deformed material at the indentation edge. Because the edge is curved, the most common measuring system, optical light microscopes, display the edge differently depending on several factors that contribute to variations in the measurement results. Studies [6–8] have shown that these influences include light intensity, incident light direction, the numerical aperture of the lens, surface roughness and the operator's subjective interpretation of the indentation edge. If each of the influence quantities can be optimized and clearly defined, then measurement of the Brinell indentation could possibly be defined. For example, the CCM-WGH is currently proposing that an optical microscope having a numerical aperture (NA) greater than 0.4 be used when measuring Brinell indentation diameters. Similar issues occur for measuring Vickers and Knoop indentations but to a lesser degree. Measurement differences can occur due to operator interpretation of the tip locations when chosen manually or due to the use of different software algorithms when determined through automated systems.

4.2. Testing cycle

The second category of parameters that significantly influences the hardness result is the testing cycle. The testing cycle describes the sequence of applying the changing levels of force on the indenter during the hardness test, as well as the measurements of indentation depth in the case of the Rockwell test. Each of the hardness tests requires periods of increasing and decreasing force applied to the indenter and periods of maintaining the force at a constant level. The testing cycle can significantly influence the indentation process and hardness result depending on the creep and strain rate behavior of test sample material. By varying the rate at which indentation occurs or the time that a force is held constant, the depth of indentation will vary and affect the hardness result. Consequently, the dwell times when the force is held constant and the application and removal of forces on the indenter must be defined.

One of the major advantages of the conventional hardness tests as an indication of material properties is that the tests can be conducted quickly. However, rapid hardness testing usually leads to less repeatability in the measurement results. The conventional hardness tests were developed to balance short test times with levels of repeatability suitable for industry's testing needs. Normally, the inclination of NMIs is to make the most stable and repeatable measurements possible. This compelled the CCM-WGH to consider defining some testing-cycle parameters at their most stable values, which were outside the limits specified by the consensus test method standards. This issue was robustly debated within the CCM-WGH.

Recognizing that variations in test parameter values will produce differences in hardness results, the CCM-WGH ultimately chose to align with the definitions specified in the consensus test method standards. At the same time, it was decided to define the testing-cycle parameter values, where possible, as single values at the most stable regions of the value ranges allowed by the standard methods, and to also specify measurement procedures where the standards are considered lacking for NMI measurements. Following this path ensures that the reference values produced by NMIs will reduce sources of measurement bias between NMIs while at the same time being aligned with industrial measurements that adhere to consensus standard test methods.

In the case of the Rockwell hardness test, NMIs have shown that indentation creep of the sample material occurs during the preliminary-force and total-force dwell times. During the recovery-force dwell time, the sample material experiences elastic recovery with a small reverse-plasticity component as shown in the expanded views of the

indentation depths in Fig. 1. The magnitude of depth change during the dwell times is directly correlated to a change in hardness value. The rate of depth change during the force dwell times is rapid at the start and diminishes over time. The CCM-WGH chose to define the dwell times to be at the longest time within the time limits of the test method standards. NMI research on the influence of dwell times on the hardness measurement result influenced ASTM and ISO committees to recommend target times within the specified Rockwell hardness standards dwell time limits that coincide with the CCM-WGH definitions.

NMIs have also shown that the rates of application and removal of the test forces can influence the hardness result. These parameters are usually specified as indentation times or indenter velocities. Applying the forces too slowly allows additional indentation creep as occurs during dwell times. Applying the forces too rapidly can affect the indentation depth due to material rate sensitivity, and for hardness machines that apply force by way of weights, it can produce force overloads due to excessive weight momentum. For the Rockwell hardness test, the hardness result is most sensitive to the rate at which the force is applied during the application of the final 20% of the total force. This is addressed in the current CCM-WGH Rockwell C scale (HRC) definition [6]. The same dwell time and force application issues occur for the Brinell, Vickers and Knoop test cycles. These test methods will undergo similar examination of the test cycle effects when developing definitions.

5. Current and future definitions

Currently in 2021, only the definition of the Rockwell C scale (HRC) has been approved by the CCM-WGH and published on the website of the International Bureau of Weights and Measures (BIPM) [9]. Even so, through the interactions, collaborations and comparisons between NMIs within the CCM-WGH, the NMI's calibration procedures have moved towards normalizing as more research is conducted and instruments are improved. New definitions of the Rockwell 15 N scale (HR15 N), Rockwell 30 N scale (HR30 N) and Rockwell 45 N scale (HR45 N) and a revised definition of the Rockwell C scale (HRC) were recently approved by the CCM-WGH and will soon be published on the BIPM website. An interesting aspect of these hardness definitions is that, once developed, they may not necessarily remain fixed. Being test method dependent measurements, the definitions will improve as the test methods improve.

References

[1] ASTM International, West Conshohocken, PA, https://www.astm.org.
[2] International Organization for Standardization, Geneva, https://www.iso.org, 2016.
[3] https://www.bipm.org/en/committees/cc/ccm/.
[4] https://www.npl.co.uk/products-services/advanced-materials/hardness-testing.
[5] J. Song, S. Low, A. Zheng, P. Gu, Geometrical Measurements of NIST Standard Reference Material Rockwell Diamond Indenters," *IMEKO 2010 TC3, TC5 and TC22 Conferences*, Metrology in Modern Context, 2010. Pattaya, Thailand.
[6] G. Barbato, S. Desogus, Problems in the Measurement of Vickers and Brinell Indentations. *Measurement*, Oct-Dec 1986, pp. 137–147, v4, No4.
[7] E. Meyer, Untersuchungen über Härteprüfung und Härte Brinell Methoden, Z. Ver. Deut. Ing. 52 (1908).
[8] R. Ellis, A. Knott, K. Herrmann, Verification of Image Analysis Systems for Measuring Brinell Indentations, 2006. *XVIII IMEKO World Congress*, Rio de Janeiro, Brazil.
[9] www.bipm.org/wg/CCM/CCM-WGH/Allowed/International_definitions/HRC_definition.pdf.

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