National Institute of Standards & Technology

Certificate

Standard Reference Material® 2812

Rockwell C Scale Hardness – High Range

Serial Number: SAMPLE

This Standard Reference Material (SRM) is a transfer standard intended for use in the calibration and verification of the performance of Rockwell hardness equipment using the Rockwell C Hardness Scale (HRC). A unit of SRM 2812 is a steel test block, nominally 64 mm in diameter and 15 mm thick, having a polished test surface with a micro-engraved circle 52 mm in diameter. Each SRM unit bears a unique serial number on the edge of the block.

Certified Hardness Values: This SRM is certified using two different certification modes: (1) the certified average hardness value over the entire test surface and (2) individually certified hardness values at specific untested locations on the test surface. The certified average hardness value (Certification Mode 1) provides the user with a comparison value for performing indirect verifications [1] of Rockwell hardness machines for the HRC scale. In comparison of user measurements with the certified average hardness, the user must account for material non-uniformity as manifest at the measurement locations chosen by the user. This non-uniformity is not included in the uncertainty for Certification Mode 1 given in this certificate. The individually certified values of hardness at specific untested locations (Certified Mode 2) provide a more complete characterization of the surface hardness of the block. The Certification Mode 2, through the certified hardness values are their uncertainties, accounts for material non-uniformity over the surface of the block.

Expiration of Certification: The certification of **SRM 2812** is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Accordingly, periodic recalibration or recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this material over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Hardness measurements leading to the certification of this SRM were performed by J.L. Fink, R.D. Jiggetts, D.R. Kelly, S.R. Low, D.J. Pitchure, and R.E. Ricker of the NIST Materials Science and Engineering Division.

Geometric verification of the Rockwell diamond indenter used by NIST in the certification of this SRM was conducted by J.F. Song and T.V. Vorburger of the NIST Semiconductor and Dimensional Metrology Division.

Statistical analysis was performed by W.S. Liggett of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Materials Science and Engineering Division

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Gaithersburg, MD 20899
Certification Issue Date: 25 April 2013
Certificate Revision History on Last Page

Certification Procedure: Several test blocks were randomly selected from each production batch and covered with hardness indentations using the NIST standardizing hardness tester and diamond indenter. The NIST standardizing hardness tester is a specially designed machine using directly loaded dead-weights for applying the required forces, and a laser interferometry displacement sensor for measuring the depth of indentation. Hardness was measured by indentation tests in accordance with the Rockwell hardness principle [1] and following the test cycle given in this certificate.

By analyzing the hardness profile of this set of test blocks, information on material uniformity was developed. NIST determined that the hardness varies across the surface of each block and varies differently from block to block. This hardness variation is due primarily to the nature of steel and the manufacturing process.

The geometry of the NIST standardizing diamond indenter was verified using a stylus instrument [2]. The indenter measurements and expanded uncertainties (at a 95 % level of confidence) are 199.06 µm ± 1.97 µm for the tip radius and 120.00° ± 0.02° for the cone angle.

Hardness measurements were then made at eight locations on the test surface of each SRM as illustrated in Figure 1. The first indentation, labeled "Seat", was made to ensure that the new test block was adequately seated on the anvil. "Seat" indentation data are potentially erroneous; thus, the resulting hardness value from this first indentation was discarded. The seven hardness tests that followed, labeled 1 through 7, were used to determine the SRM certified hardness values for that SRM.

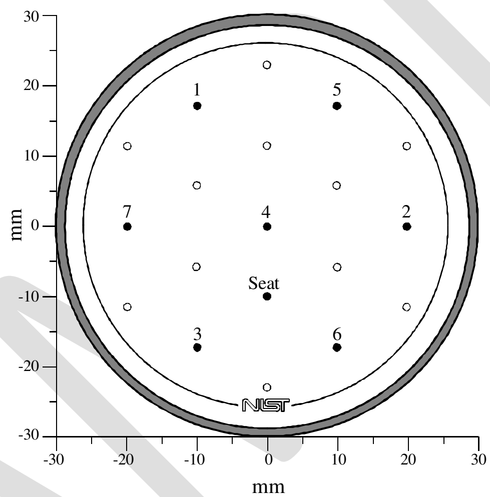

Figure 1. Test surface of SRM indicating the location and sequence of certification indentations (shown as black dots), and illustrating the appearance of the engraved circle and NIST logo. The labels 1 through 7 and "Seat" shown on the figure indicate the indentation sequence used in certification.

Certification Mode 1, Certified Average Hardness Value: This value is an estimate of the average hardness across the test surface of the block. Hardness varies slightly across the surface due to material non-uniformity and can be modeled by a mathematical function, a semivariogram (see "Discussion"). The average hardness value is defined as that hardness function integrated over the test block surface divided by the surface area.

Certified Average Hardness Value: SAMPLE HRC ± SAMPLE HRC

Uncertainty of the Certified Average Hardness Value: The expanded uncertainty, U, in the certified average hardness was calculated using $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the JCGM Guide [3], and $k = 2$ is the coverage factor. The value of u_c represents the combined effect of the individual sources of uncertainty listed in Table 1. These sources include uncertainties due to deviations of the NIST standardizing hardness tester and diamond indenter from the defined requirements for the Rockwell hardness test (forces, depth measurement, test cycle, and indenter shape) [1]. The value of u_c is calculated as follows:

$$u_c = \sqrt{\sigma_1^{\,2} + \sigma_2^{\,2} + \sigma_3^{\,2} + \sigma_4^{\,2}} \tag{1}$$

Table 1. Sources of Uncertainty for the Certified Average Hardness Value

Uncertainty Source		Standard Uncertainty (HRC)
σ_1	Material Uniformity and Measurement Repeatability	± SAMPLE
σ_2	Day-to-Day Reproducibility	± SAMPLE
σ_3	NIST Standardizing Hardness Tester	± SAMPLE
σ_4	NIST Standardizing Indenter	± SAMPLE
u_c	Combined Standard Uncertainty	± SAMPLE
U	Expanded Uncertainty	± SAMPLE

The expanded uncertainty, U, provides an indication of the accuracy of NIST's estimate of the true value of the average hardness of this SRM. The uncertainty should not be interpreted as the range in expected hardness values that would be measured across the test surface, nor is it a limit of acceptable hardness values for verifying Rockwell hardness equipment. Similarly, σ_1, σ_2, σ_3, and σ_4 are uncertainty components that contribute to the overall uncertainty in the certified average hardness value and are not ranges of expected hardness results due to each source of uncertainty.

Certification Mode 2, Individually Certified Hardness Values for Specific Locations: This certification mode provides the user with individually certified hardness values at 11 untested locations on the SRM. The certified values at each location, defined in x, y coordinates and labeled with the letters A through K, are given in Table 2 and illustrated in Figure 2.

Table 2. Individually Certified Hardness Values for Specific Test Block Locations

Location	x (mm)	y (mm)	Hardness (HRC)
A	0	23	SAMPLE ± SAMPLE
B	20	12	SAMPLE ± SAMPLE
C	20	– 12	SAMPLE ± SAMPLE
D	0	– 23	SAMPLE ± SAMPLE
E	– 20	– 12	SAMPLE ± SAMPLE
F	– 20	12	SAMPLE ± SAMPLE
G	0	12	SAMPLE ± SAMPLE
H	10	6	SAMPLE ± SAMPLE
I	10	– 6	SAMPLE ± SAMPLE
J	– 10	– 6	SAMPLE ± SAMPLE
K	– 10	6	SAMPLE ± SAMPLE

The x, y coordinate system is such that location $x = 0$, $y = 0$ is at the block center (NIST indentation 4) and oriented with the NIST logo at the bottom of the block as illustrated in Figure 2.

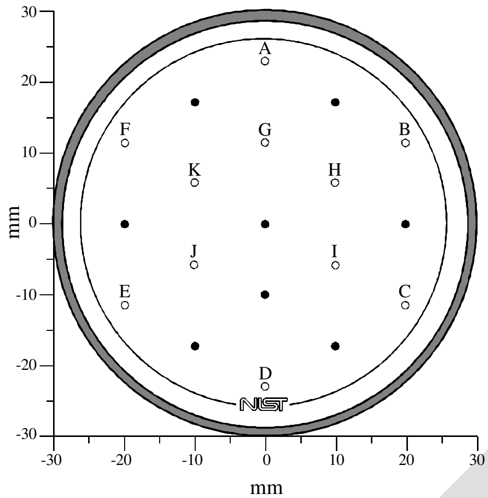

Figure 2. Test surface of SRM illustrating the locations (shown as open circles labeled A through K) of individually certified hardness values given in Table 2.

Uncertainty of Individually Certified Hardness Values: The uncertainties in the individually certified hardness values for the locations reported in Table 2 are calculated using the same sources of uncertainty as used for the certified average hardness values given in Table 1. A difference occurs in σ_1, the uncertainty component due to block uniformity and measurement repeatability. The remaining uncertainty components σ_2, σ_3, and σ_4 are the same for both types of hardness certification. These four uncertainty components are given in Table 3 at a level of one standard deviation. These components are combined to calculate the standard uncertainty, u_c, to which the coverage factor $k = 2$ is applied to obtain the expanded uncertainty, U.

For the individually certified hardness values, the expanded uncertainty, U, provides an indication of the accuracy of the best estimate of the true value of the hardness at that specific location on the SRM. Similarly, σ_1, σ_2, σ_3, and σ_4 are uncertainty components that contribute to the overall uncertainty in the certified hardness value at each specific location.

Table 3. Sources of Uncertainty for Hardness Values at Each of the Individual Locations Given in Table 2

Uncertainty Source		Standard Uncertainty Locations A through F (HRC)	Standard Uncertainty Locations G through K (HRC)
σ_1	Material Uniformity & Repeatability	± SAMPLE	± SAMPLE
σ_2	Day-to-Day Reproducibility	± SAMPLE	± SAMPLE
σ_3	NIST Standardizing Hardness Machine	± SAMPLE	± SAMPLE
σ_4	NIST Standardizing Indenter	± SAMPLE	± SAMPLE
u_c	Combined Standard Uncertainty	± SAMPLE	± SAMPLE
U	Expanded Uncertainty	± SAMPLE	± SAMPLE

Discussion: To illustrate the effect of the material non-uniformity, the test surface of the SRM block should be considered a collection of possible measurement locations. The metal at each location will have a unique hardness value that differs slightly from the hardness value at any other location. If it were possible to determine the hardness at every location, the collection of hardness values would be found to vary over a range, and an average hardness value for the entire test surface could then be calculated. The extent of this range defines the non-uniformity in hardness for the test surface. The smaller the range in hardness values, the more uniform is the block. Thus, for this SRM, the hardness values at individual locations will vary over a range that extends both above and below the average hardness value of the test surface.

As a consequence of this hardness non-uniformity, a single certified hardness value does not completely describe the hardness of the test surface. Thus, two types of hardness certifications are provided in this certificate: (1) the certified average hardness (Certification Mode 1) and (2) individually certified hardness values (Certification Mode 2) at untested locations. The certifications are based on the seven NIST hardness measurements and on modeling hardness as a random function across the test surface. These two types of certifications define the hardness of the test block in distinctly different ways. The user must keep this in mind when using these certified values and the associated uncertainties.

In general, the closer two measurements are made, the closer the hardness values will be to each other, limited by minimum spacing considerations (see "Indentation Spacing"). The method used to predict hardness values at untested locations is based on a geostatistics formula [4] that models the hardness across the surface of a block as a random function described by a semivariogram. The semivariogram is a mathematical model that describes how the measured hardness difference between any two test block locations relates to the physical spacing that separates the two locations. In statistical terms, this semivariogram gives one half the variance of the hardness difference between any two locations on the test block. Thus, the square root of twice the semivariogram is the standard deviation of this difference.

For this SRM, the semivariogram is given by a simple function of Euclidean distance. Consider two points, s_i and s_j, separated by the distance, d (in mm), where

$$d = \sqrt{(x_i - x_j)^2 + (y_i - y_j)^2} \tag{2}$$

The semivariogram is given by

$$\gamma(s_i - s_j) = \begin{cases} 0 & \text{if } d = 0 \\ c_0 + c_e\left[1 - \exp\left(-d/a_e\right)\right] & \text{if } d \neq 0 \end{cases} \tag{3}$$

where c_0, c_e, and $1/a_e$ are given in Table 4. In addition to the semivariogram, the calculation of certified hardness values required the seven hardness readings obtained by NIST, given in Table 5.

Table 4. Semivariogram Coefficients that Describe Uniformity and Repeatability

Coefficients	Values
c_o	SAMPLE
c_e	SAMPLE
$1/a_e$	SAMPLE

Table 5. NIST Hardness Measurements for Individual Test Block Locations

Location	x (mm)	y (mm)	Hardness Value (HRC)
1	–10	17	SAMPLE
2	20	0	SAMPLE
3	–10	–17	SAMPLE
4	0	0	SAMPLE
5	10	17	SAMPLE
6	10	–17	SAMPLE
7	–20	0	SAMPLE

The certified average hardness value is based on the seven NIST hardness measurements and on the hardness prediction function discussed above. This average value is defined as the hardness function integrated over the test block surface divided by the surface area, and as such, is the average of the predicted hardness values for *all* test surface locations, and *not* the arithmetical average of the seven NIST measurements. However, since the locations chosen for the seven NIST measurements provide a good representation of the surface, the certified average hardness is close in value to the arithmetical average. The NIST certified average hardness value is only somewhat analogous to the certified value assigned to a commercially calibrated hardness test block that *is* typically calculated as the arithmetical average of several calibration measurements.

This certificate also provides individually certified hardness values for 11 specific untested locations on the test surface of the SRM, given in Table 2. These certified hardness values and their associated uncertainties were calculated using a mathematical formula [4], which includes the semivariogram given above and the three coefficients given in Table 4. All that is required to perform these calculations are the values and locations for the seven NIST measurements given in Table 5, and the *x*, *y* coordinates of the locations for which hardness values are to be determined. Using this same formula, additional certified hardness values at other untested locations may be calculated by the user [5,6]. The formulas may also be used for calculating various averages of the hardness at two or more arbitrary locations.

Traceability: This SRM can be used to demonstrate that a user's measurements are traceable to the HRC scale as determined by NIST; however, the procedures required to demonstrate traceability are varied depending on the user's needs. In general, acceptable transfer of the NIST hardness values to the user can be accomplished in three ways: (1) *verification within tolerances* - verifying that the user's measurements are in agreement with NIST certified values within acceptable tolerance limits; (2) *verification within uncertainties* - verifying that the user's measurements are in agreement with NIST certified values within the uncertainty ranges of the user's and NIST's measurements; and (3) *calibration* - calibration of the performance of the user's hardness machine to the NIST certified values by making appropriate mathematical corrections, and then verifying that the user's corrected values are in agreement with NIST certified values within the uncertainties of the user's and NIST's measurements. Each of the three methods requires differing levels of effort by the user to determine the accuracy in his/her measurements.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This SRM is made of steel, and thus is susceptible to scratches, dents, and other mechanical damage, particularly on the top and bottom surfaces, as well as being susceptible to corrosion from moisture and reaction to skin oils. The SRM is wrapped in corrosion-inhibiting paper and packaged in a protective wooden box. The user should wear protective gloves, such as lint-free cotton or powder-free latex gloves, while handling the SRM. If gloves are not worn, skin oils should be removed by cleaning the SRM, see "Cleaning the SRM" below. The SRM should be held only by the round edge to avoid excessive contact with the top and bottom surfaces.

Storage: When not in use, the SRM should be rewrapped and stored in the original box or in a manner having equivalent or better mechanical and corrosion protection. The SRM storage area should be maintained at 23 °C ± 2 °C (73 °F ± 5 °F) with a relative humidity of 50 % or less.

Use: The SRM is intended for use with all equipment capable of conducting an HRC indentation hardness test. The HRC hardness test is defined as requiring the use of a Rockwell spheroconical diamond indenter and the following test forces: a preliminary force of 98 N (10 kgf) and an additional test force of 1373 N (140 kgf) that, when combined with the preliminary force, generates the total test force of 1471 N (150 kgf). The Rockwell hardness tester should be set up and operated in accordance with the recommendations of the manufacturer and as required by applicable test method standards. All hardness measurements must be made within the circle engraved on the test surface of the SRM. In order to perform calibration or verification measurements over the full usable range of the HRC scale, this SRM may be used in conjunction with SRM 2810 Rockwell C Scale Hardness - Low Range and SRM 2811 Rockwell C Scale Hardness - Mid Range.

Test Environment: The hardness tester to be calibrated or verified should be stored in a temperature and humidity-controlled environment maintained at 23 °C ± 2 °C (73 °F ± 5 °F) and a relative humidity of 50 % or less and that the environment has been calibrated or verified to maintain those conditions. The tester must also be in a location that is free from shock or vibration that could affect the hardness measurements.

Anvil: Use of a flat anvil is recommended (i.e., an anvil that can support the SRM for indentation anywhere on its test surface). However, when calibrating or verifying a hardness tester, the same anvil must be used with the SRM that will be used on the tester after the calibration or verification.

Cleaning the Anvil and Indenter: The tester anvil and indenter diamond tip should be thoroughly cleaned using the recommendations from the manufacturer. In the absence of cleaning instructions from the manufacturer, it is recommended that the anvil and indenter be cleaned with ethyl alcohol and dried using a lint-free paper towel or cloth. Then the surfaces should be blown clean of dust using filtered air, such as from a commercial compressed air can or bottle. **DO NOT** blow clean by mouth.

Cleaning the SRM: Prior to use, the SRM should be cleaned. The SRM may be cleaned by gently wiping the top and bottom surfaces with a clean cotton or a non-abrasive cloth thoroughly wetted with ethyl alcohol. The metal surfaces should immediately be dried using a soft lint-free paper towel or cloth before the alcohol evaporates. There should be no residue remaining on the top and bottom surfaces. The alcohol cleaning should be followed by blowing the surfaces clean of dust with filtered air. The top and bottom surfaces should **NOT** be touched after cleaning.

Seating the Anvil and Indenter: Prior to making hardness indentations on the SRM, the tester anvil and indenter must be seated. This can be accomplished by performing Rockwell hardness tests on a material using the highest test force the tester is capable of applying. The seating tests should be repeated until successive measurement values show no trend of increasing or decreasing hardness.

Placement of the SRM on the Anvil: Immediately before placing the SRM on the anvil, the top surface of the anvil and the bottom surface of the SRM should be cleaned as described above. Once clean, the SRM should be placed immediately on the anvil. The top test surface of the SRM should be blown free of dust prior to testing and occasionally during use. The SRM should be slid several times back and forth over the surface of the anvil to help seat the block on the anvil. Any time the SRM is lifted from and replaced on the anvil, the procedure described above in this paragraph should be repeated.

Preliminary Indentation: When a flat anvil is used, at least one preliminary HRC test should be performed at any location on the test surface of the SRM. The preliminary test will help seat the SRM on the anvil. The measured hardness value of the preliminary test should not be used. A preliminary indentation is not necessary when using a spot anvil.

Indentation Location: The user is cautioned not to make any indentation such that it contacts a previous indentation, the engraved circle, or the NIST logo; as doing so may damage the indenter. No indentation should be made outside the engraved circle or on the bottom surface of the block.

Indentation Spacing: Rockwell hardness measurements may be influenced by nearby previous indentations. For this SRM, a hardness measurement will not be measurably affected by an adjacent Rockwell C Scale indentation that is located at a distance of 5 mm or greater, center-to-center spacing, from the measurement location. It is recommended that no indentation should be made closer than 3 mm, center-to-center spacing, from a previous indentation. For hardness measurements within 3 mm to 5 mm distance of only one single previous indentation, the resulting HRC value may be increased as much as 0.05 HRC. Measurements made within 3 mm to 5 mm distance from *multiple* previously made indentations will produce an even higher elevation in HRC. The user should consider this possible elevation of hardness when making measurements on this SRM. Table 6 lists the effect of indentation-to-indentation spacing for this SRM. In addition to this indentation-to-indentation spacing restriction, no HRC measurement should be made within 1 mm of the engraved circle or the NIST logo.

Table 6. Effect of Indent Spacing

Distance Between Indentations (Indentation Center-to-center)	Effect on Hardness
≥5 mm	No interaction
≥3 mm and <5 mm	Increase in hardness of up to 0.05 HRC
<3 mm	Not recommended

Test Cycle: This SRM unit has been certified by performing Rockwell C scale hardness tests using a specific test cycle. The HRC test cycle may be characterized by specifying four test cycle parameters that have been determined to have a significant influence on the measurement results. These parameters are defined as follows:

> ***Preliminary Test Force Dwell Time*** – the time period beginning when the preliminary force is fully applied and ending when the first baseline measurement of indenter depth is taken.
>
> ***Total Force Dwell Time*** – the time period during which the total force is fully applied.
>
> ***Recovery Dwell Time*** – the time period beginning when the additional test force is fully removed returning the applied force to the preliminary force level and ending when the final measurement of indenter depth is taken.
>
> ***Additional Force Application Time*** – the time period during the application of the additional force. This parameter may also be specified in terms of the indenter velocity during this time period.

To minimize the uncertainty in the hardness measurement, a test cycle should be used that replicates, as closely as possible, the SRM certification test cycle parameters. Deviations from the SRM test cycle in dwell times or force application rate will result in measured hardness values that deviate from the certified values.

The test cycle parameters used for the certification of this SRM are given in Table 7 and illustrated in Figure 3.

Table 7. Specifications for the SRM Test Cycle Parameters

Parameter	Value	NIST Tolerance
Preliminary Test Force Dwell Time	3 s	± 0.1 s
Total Force Dwell Time	5 s	± 0.1 s
Elastic Recovery Dwell Time	4 s	± 0.1 s
Indenter Velocity	40 µm/s constant	± 2 µm/s

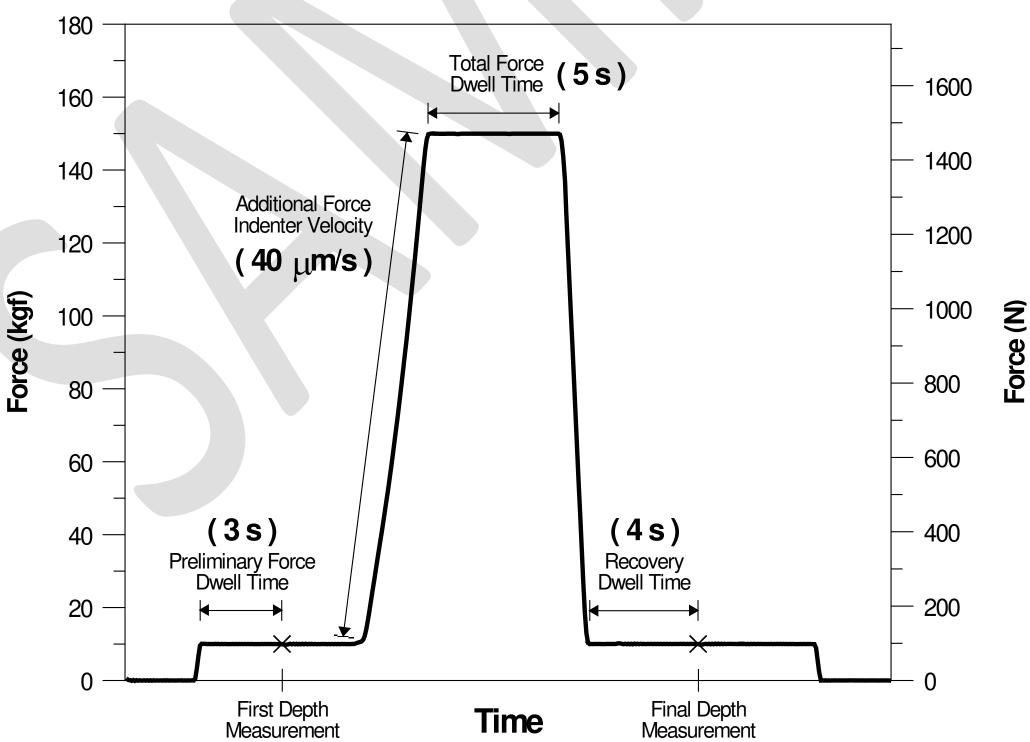

Figure 3. Illustration of the parameters of the HRC Test Cycle used in the certification of the SRM.

REFERENCES

[1] ASTM E18; *Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials*; Annu. Book ASTM Stand.

[2] Song, J.F.; Rudder, F.F. Jr.; Vorburger, T.V.; Smith, J.H.; *Microform Calibration Uncertainties of Rockwell Diamond Indenters*; J. Res. Natl. Inst. Stand. Technol., Vol. 100, pp. 543–561 (1995).

[3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2013); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed Apr 2013).

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[5] Liggett, W.S.; Low, S.R.; Pitchure, D.J.; Song, J.; *Capability in Rockwell C Scale Hardness*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 511–533 (2000).

[6] Low, S.R.; *NIST Recommended Practice Guide: Rockwell Hardness Measurement of Metallic Materials*; NIST Special Publication 960-5; U.S. Government Printing Office: Washington, DC (2001); available at http://www.nist.gov/manuscript-publication-search.cfm?pub_id=853006 (accessed Apr 2013).

Certificate Revision History: 25 April 2013 (Updated document format for selected units; corrected minimum measurement spacing requirement in text, editorial changes) 20 March 2013 (Updated document format; added new units; editorial changes); 15 November 2011 (Updated certification period; editorial changes); 08 June 2011 (Updated certification period; editorial changes); 13 June 2006 (Updated certification period; editorial changes); 26 May 2005 (Updated certification period; editorial changes); 10 June 2003 (Updated certification period; editorial changes for selected units); 17 May 2002 (Editorial changes); 16 October 2001 (Changed in the uncertainty values Tables 1, 2, 3, and the semivariogram coefficients in Table 4, in addition to editorial changes); 08 December 1997 (Original certificate date).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.