

CALIBRATION REPRODUCIBILITY TEST FOR NIST's No. 3581 STANDARD ROCKWELL DIAMOND INDENTER

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Abstract: Standard Rockwell diamond indenters play an important role for a worldwide unified Rockwell hardness scale. In 1994, the National Institute of Standards and Technology (NIST) established a Microform Calibration System with sufficiently small calibration uncertainty for the calibration of standard Rockwell indenters. In 1995, NIST established a group of Rockwell diamond indenters characterized both by high geometrical uniformity and hardness performance uniformity. One of them, No. 3581, was selected as the NIST's primary Rockwell diamond indenter for the calibration of NIST's Standard Reference Material (SRM) Rockwell C hardness blocks. This indenter was recalibrated in 1997 and 2005. The calibration results showed high stability for the microform geometry of the NIST's standard Rockwell diamond indenter, as well as high calibration reproducibility for the NIST's Microform Calibration System.

Keywords: Rockwell hardness, HRC, diamond indenter, traceability, reproducibility, microform calibration.

1. INTRODUCTION

Rockwell hardness (HR) is the most widely used mechanical testing method for metal products. Rockwell hardness scales are empirical, and as such are defined by reference standards (standard testing machine and indenter) and reference testing conditions. A Rockwell hardness scale is established by the performance of a standard diamond indenter (for the HRC, HRD, HRA, HR45N, HR30N and HR15N scales) using a standard testing machine and a standardized testing cycle [1]. Recent developments in standard hardness machines and microform calibration techniques have made it possible to establish a worldwide unified Rockwell hardness scale with metrological traceability [2]. This includes both the establishment of the reference standards (standard machine and diamond indenter) traceable to SI units of force, time, and length; and the establishment of the reference testing conditions (a standardized common testing cycle) based on an international agreement.

Standard Rockwell diamond indenters play an important role for a worldwide unified Rockwell hardness scale. In 1994, the National Institutes of Standards and Technology (NIST) established a Microform Calibration System based on a stylus instrument and a set of calibration and check standards; and developed calibration and uncertainty procedures for the microform calibration of Rockwell

diamond indenters [3]. The expended calibration uncertainties were sufficiently small for the calibration of the standard Rockwell indenters. In 1995, a group of standard Rockwell diamond indenters were calibrated and characterized both by high geometrical uniformity and hardness performance uniformity. One of them, No. 3581, was selected as the NIST primary standard for the calibration of NIST's SRM 2810 to 2812 Rockwell C hardness blocks [1, 4]. After about three years of use and more than 3000 indentations, this indenter was recalibrated in 1997. The calibration results showed high stability for the microform geometry and hardness performance of the NIST's primary standard Rockwell diamond indenter, and high calibration reproducibility for NIST's Microform Calibration System [5].

In 2004, the Advanced Measurement Laboratory (AML) was constructed at NIST. The microform calibration laboratory was moved to this new building. Meanwhile, several upgrades of the hardware and software of the measurement system were completed. In order to check the calibration reproducibility for the Microform Calibration System and the stability of the NIST standard Rockwell diamond indenter, the No. 3581 indenter was recalibrated in November 2005 using the same calibration and check standards and the same calibration procedure, but the calibration was performed by a different person than the 1994 and 1997 calibrations. The purpose was to examine whether or not there is a significant difference in the calibration results when the calibration system, calibration and check standards and calibration procedure are used by a different person; to test the possible bias between different operators; and to ensure that reproducible calibration results for NIST's primary Rockwell diamond indenter can be achieved not only from time to time, but also from one operator to another, as long as the calibration procedures are followed.

2. CALIBRATION TRACEABILITY FOR NIST STANDARD ROCKWELL INDENTERS

The microform geometry calibrations of the Rockwell diamond indenter include those for the 200 μm tip radius and form deviation from the radius, the 120° cone angle and cone flank straightness, and the holder axis alignment error.

There are two grades of Rockwell indenters specified in Rockwell hardness test method standards published by the International Organization for Standardization (ISO) [6, 7] and ASTM-International [8]. Working grade indenters

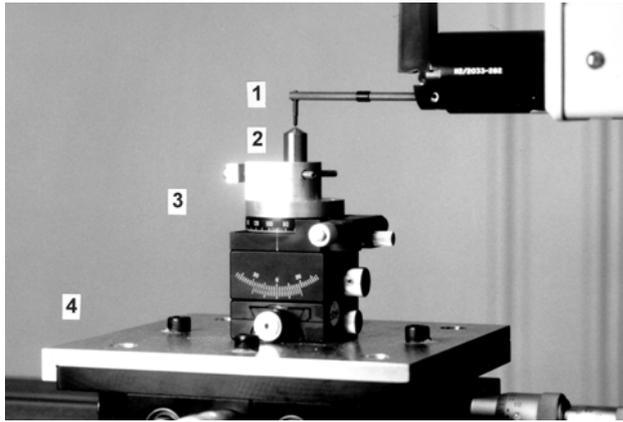


Fig. 1. NIST Microform Calibration System for Rockwell diamond indenters: (1) stylus tip; (2) Rockwell indenter; (3) rotary stage; (4) x-y stage.

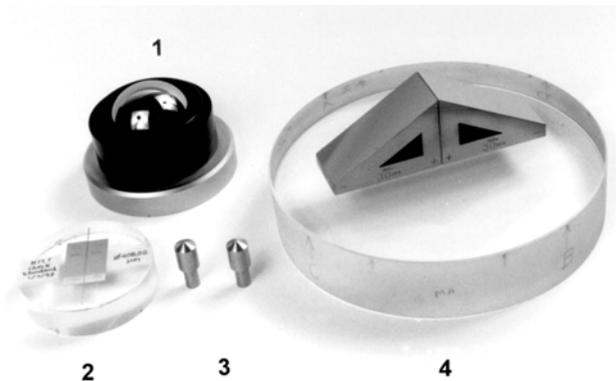


Fig. 2. Calibration and check standards: (1) 22 mm radius calibration ball; (2) 200 μm radius standard wire; (3) 200 μm radius ruby balls mounted on the tip of the Rockwell indenter-shaped holders; (4) 120° angle gauge block.

specified in ISO 6508-2[6] and ASTM E18 [8] are used for ordinary hardness tests. Calibration grade indenters specified in ISO 6508-3 [7] and ASTM E18 [8] are used for calibrations of secondary Rockwell hardness block reference standards. The geometric tolerances are shown in Table 1.

Geometrical nonuniformity and calibration uncertainty for the Rockwell indenters take a major part in the uncertainty budget of Rockwell hardness tests [9]. At NIST, a Microform Calibration System was established in 1994 based on a commercial stylus instrument with an x-y-rotary stage (see Fig. 1). A set of calibration and check standards (see Fig. 2) was developed for instrument calibration and to ensure measurement traceability to the SI unit of length. These standards include a calibration ball with 22 mm nominal radius, a standard wire and two ruby balls with 200 μm nominal radii, and a 120° angle gauge block. Three methods were used for calibrating the 2 μm nominal tip radius of the stylus instrument and for making measurement corrections [10].

The measurement procedure is as follows. Before calibrating a Rockwell diamond indenter, the instrument was calibrated with the 22 mm radius standard ball. The calibration was certified by measuring the check standard artifacts including the standard wire, the ruby balls if necessary, and the angle gauge block. After this, the Rockwell indenter was calibrated at nine sections 40° apart. In each section, the stylus was first crowned on the top point of the diamond indenter. Then a 1.2 mm long trace was made across the crown of the indenter and 4800 data points were collected. By windowing on the central $\pm 100 \mu\text{m}$ part of the range and using least squares arc fitting, the least squares radius and profile deviation from the least squares fitted radius were determined. By windowing on the remaining left and right portions of the trace, located from $-450 \mu\text{m}$ to $-100 \mu\text{m}$ in the left and from $+100 \mu\text{m}$ to $+450 \mu\text{m}$ in the right, and using the least squares line fitting algorithms, the indenter cone angle and cone flank straightness error were determined. The indenter holder axis alignment error was calculated from the cone angle measurements at nine sections 40° apart. The surface roughness can also be measured by using the same traced profile on the cone angle by selecting the appropriate filter cutoff. The last step in the procedure is to check the instrument calibration once again, and check the measurements by remeasuring the check standards including the standard wire and angle gauge block.

An uncertainty procedure was developed for calculating the calibration uncertainties of the Rockwell diamond indenters. The expanded uncertainty ($k = 2$) of the measurement system is $\pm 0.4 \mu\text{m}$ for the 200 μm least squares radius calibrations, and $\pm 0.01^\circ$ for the 120° cone angle calibrations. Detailed information can be found in Ref. 3. These expanded uncertainties are sufficiently small for the geometry calibrations of the standard Rockwell diamond indenters. In addition, the geometric form errors of the Rockwell indenters including the profile deviations from the least squares radius Pp and Pv [11], the cone flank straightness Pt [11], the surface roughness Ra [11] and the holder axis alignment error are also measured.

3. CALIBRATION REPRODUCIBILITY TEST FOR NIST's No. 3581 STANDARD ROCKWELL DIAMOND INDENTER

In order to maintain the long term stability for the National Rockwell Hardness Scale, the calibration and check standards for the Microform Calibration System are periodically calibrated by the dimensional metrology laboratory of NIST. NIST's primary Rockwell diamond indenter is also calibrated to ensure the stability of the standard Rockwell indenter and the reproducibility of the Microform Calibration System.

The No. 3581 Rockwell diamond indenter was first calibrated in 1995 and used for the calibration of NIST SRM Rockwell C hardness blocks. From 1995 to 1997, about 300 NIST SRM blocks were calibrated using this indenter, with a total of more than 3000 indentations. In October 1997, this NIST's primary indenter was recalibrated using the same Microform Calibration System. In November 2005, after the Microform Calibration System

was moved to the new building, and the measurement system was upgraded, the No. 3581 indenter was calibrated once again. The results of the three calibrations from 1995, 1997 and 2005 are shown in Table 1.

The variation of the calibration results from 1995 to 2005 were within the range of the expanded measurement uncertainties. For example, the mean least squares radius was calibrated as $(199.06 \pm 1.97) \mu\text{m}$ in 1995, $(199.24 \pm 1.19) \mu\text{m}$ in 1997, and $(199.11 \pm 1.30) \mu\text{m}$ in 2005. The mean cone angle was calibrated as $(119.995 \pm 0.020)^\circ$ in 1995, $(120.012 \pm 0.017)^\circ$ in 1997, and $(119.987 \pm 0.020)^\circ$ in 2005. The profile deviations from the least squares radii, including the maximum peak height Pp , the minimum valley depth Pv , and the cone flank straightness Pt also show no significant changes (see Table 1). These comparison results have demonstrated a long-term stability for both the microform geometry of the NIST primary Rockwell indenter, as well as long-term measurement reproducibility for the NIST Microform Calibration System. NIST is currently working with diamond manufacturers to produce SRM Rockwell hardness diamond indenters to support U.S. industry and international Rockwell hardness standardization.

4. SUMMARY

The NIST's No 3581 primary Rockwell diamond indenter was established in 1995, and recalibrated in 1997 and 2005. The calibration results showed that:

- The Rockwell diamond indenter can hold its microform geometry for many years and after thousands of hardness indentations, as long as the indentation operations are carried out correctly;
- NIST's primary Rockwell diamond indenter has also shown a long term stability;
- The NIST Microform Calibration System has shown long term calibration reproducibility within the range of the reported expanded measurement uncertainties for the calibration of standard Rockwell indenters. It has demonstrated a capability to support the long term stability of the National Rockwell Hardness Scales.
- The highly reproducible calibration results can be achieved by different operators, as long as the calibration procedures are followed.

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Table 1. Calibration results for NIST's No. 3581 Rockwell diamond indenter in 1995, 1997 and 2005. Uncertainties are reported for $k = 2$.

Microform Geometry Components	Tolerances		Calibration Results		
	Working Grade (ISO 6508-2:2005 ASTM E18-05)	Calibration Grade (ISO 6508-3:2005, ASTM E18-05)	4/11/1995	10/29/1997	11/5/2005
1. Spherical Radius					
1a. Mean	$200 \pm 10 \mu\text{m}$	$200 \pm 5 \mu\text{m}$	$199.06 \pm 1.97 \mu\text{m}$	$199.24 \pm 1.19 \mu\text{m}$	$199.11 \pm 1.30 \mu\text{m}$
1b. Maximum Variation	$200 \pm 15 \mu\text{m}$	$200 \pm 7 \mu\text{m}$	Max. = $200.70 \mu\text{m}$ Min. = $197.65 \mu\text{m}$	Max. = $201.58 \mu\text{m}$ Min. = $197.41 \mu\text{m}$	Max. = $201.67 \mu\text{m}$ Min. = $196.97 \mu\text{m}$
1c. Profile Deviation	$< 4 \mu\text{m}^{(1)}, \pm 2 \mu\text{m}^{(2)}$	$< 2 \mu\text{m}^{(1)}, \pm 2 \mu\text{m}^{(2)}$	$Pp = 0.40 \mu\text{m}$ $Pv = 0.29 \mu\text{m}$	$Pp = 0.45 \mu\text{m}$ $Pv = 0.33 \mu\text{m}$	$Pp = 0.43 \mu\text{m}$ $Pv = 0.33 \mu\text{m}$
2. Cone Angle					
2a. Mean	---	$120\text{E} \pm 0.1\text{E}^{(1)}$	$119.995^\circ \pm 0.020^\circ$	$120.012^\circ \pm 0.017^\circ$	$119.987^\circ \pm 0.020^\circ$
2b. Maximum Variation	$120\text{E} \pm 0.35\text{E}$	$120\text{E} \pm 0.17\text{E}^{(1)}$ $120\text{E} \pm 0.1\text{E}^{(2)}$	Max. = 120.010° Min. = 119.984°	Max. = 120.038° Min. = 119.974°	Max. = 120.018° Min. = 119.949°
2c. Cone Flank Straightness	$< 2 \mu\text{m}^{(1)}$	$< 0.5 \mu\text{m}^{(1)}$	$Pt = 0.42 \mu\text{m}$	$Pt = 0.49 \mu\text{m}$	$Pt = 0.49 \mu\text{m}$
3. Holder Axis Alignment	0.5E	0.3E	0.08°	0.022°	0.13°
4. Surface Finish					
4a. Roughness Mean	---	---	---	$Ra = 0.0035 \mu\text{m}$	---
4b. Max. Surface Roughness	---	---	---	$Ra = 0.0036 \mu\text{m}$	---

⁽¹⁾ Specified in ISO Standard [6, 7]; ⁽²⁾ Specified in ASTM Standard [8].