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GEOMETRIC MEASUREMENT COMPARISONS FOR ROCKWELL DIAMOND INDENTERS

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Abstract − In the uncertainty budget of Rockwell C hardness (HRC) tests, geometric error of the Rockwell diamond indenter is a major contributor. The geometric calibration of Rockwell diamond indenters has been a key issue for Rockwell hardness standardization. The National Institute of Standards and Technology (NIST) developed a microform calibration system based on a stylus instrument for the geometric calibration of Rockwell diamond indenters. Using that system, a NIST master standard Rockwell diamond indenter No. 3581 was established in 1995, by which approximately 300 standard reference material (SRM) Rockwell hardness blocks of HRC scale were calibrated for implementation of the HRC scale in the United States. This indenter has been re-calibrated in 1997, 2005 and 2007. The calibration results have shown both high stability for the NIST standard Rockwell diamond indenter and high reproducibility for the NIST microform calibration system. After more than fifteen years of service, the stylus instrument was recently replaced by a new one. In order to test the measurement agreement between the two stylus instruments, another NIST master Rockwell diamond indenter No. 101 has been recently calibrated by the new instrument and the calibration result shows good agreement with the previous results.

Keywords: Rockwell C hardness, diamond indenter, microform calibration

1. INTRODUCTION

Rockwell hardness is the most widely used mechanical testing method for metal products. The Rockwell hardness scales are empirical, and as such are defined by reference standards (standard testing machine and indenters) and reference testing conditions [1]. The Rockwell hardness C scale (HRC) for testing steel uses a spheroconical diamond indenter having a 120° cone angle and a 200 µm radius spherical tip. In the uncertainty budget of HRC tests, geometric error of the Rockwell diamond indenter is a major contributor [2]. International comparisons have shown that, when a "common indenter" is used, the measured hardness variation range is much less than that observed when using different national indenters [3].

2. NIST MICROFORM CALIBRATION SYSTEM FOR ROCKWELL DIAMOND INDENTERS

In order to establish a worldwide unified Rockwell hardness scale, it is important to calibrate the geometric parameters of the Rockwell diamond indenter with sufficiently small calibration uncertainties, and to maintain the long term calibration reproducibility. The NIST microform calibration system was developed for this purpose. It is based on a commercial stylus instrument with a stacked x-y-rotary stage that holds the indenter in a vertical position with the tip pointing upwards. A diamond stylus with a nominal radius of 2 µm traces over the top surface of the Rockwell diamond indenter. The x-y stage is used for crowning the stylus at the peak of the Rockwell indenter tip. The rotary stage is used to rotate the diamond indenter from one section to the other [4].

A set of calibration and check standards is used for instrument calibration and measurement quality control. These include a standard ball with a nominal radius of 22 mm, a standard wire with a nominal radius of 200 µm, and a 120° gauge block. The standard ball, provided by the instrument maker, is used for instrument calibration. The standard wire is used for checking measurements of the 200 µm radius of the Rockwell indenter. The 120° gauge block is used for checking measurements of the 120° cone angle of the Rockwell indenter. All the standards were calibrated at NIST and are traceable to the SI (*Systeme International d'Unites*, or International System of Units) unit of length – the meter.

Before the calibration of a Rockwell diamond indenter, the instrument is calibrated by measuring the 22 mm radius standard ball. The calibration is then verified by measuring the standard wire and angle gauge block. After this, the Rockwell indenter is calibrated by taking nine cross-sectional profiles rotationally separated by 40° across the tip. Translation of the x-y stage is used to ensure that the stylus is crowned on the peak of the Rockwell indenter for each profile. Then the stylus traces a 1.2 mm (or ± 0.6 mm) length across the crown of the indenter, in which 9600 data points are collected. By windowing on the central ± 100 µm part of the trace (see Figure 1) and using a least squares arc fitting algorithm, the least squares radius and profile deviation from the fitted radius are determined. By windowing on the remaining left and right portions of the

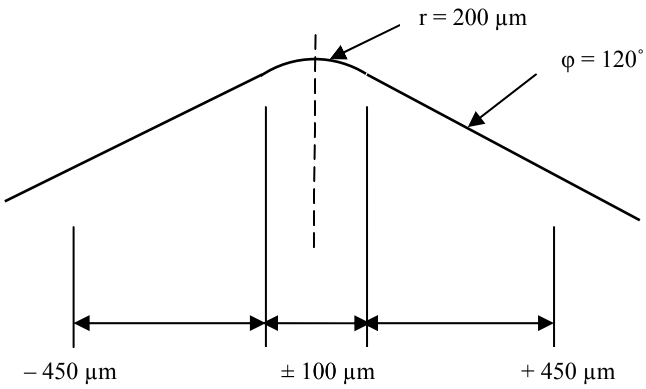

Figure 1. Three windows for data fitting of tip radius and profile deviations at ± 100 µm of the center and for cone angle and cone flank straightness fitting at ± (100 to 450) µm of the left and right.

trace, located from – 450 µm to – 100 µm on the left and from + 100 µm to + 450 µm on the right (Figure 1), and using the least squares line fitting algorithm, the indenter cone angle and cone flank straightness error are determined. The indenter holder axis alignment error is also calculated from the cone angle measurements. The surface roughness can be measured on the cone surface of the traced profile by selecting the appropriate filter cut-off. The last step in the calibration procedure is to check the measurements by re-measuring the standard wire and angle gauge block check standards.

The expanded measurement uncertainties (k = 2) for the calibrations of Rockwell diamond indenters are ± 0.3 µm for the 200 µm nominal tip radius calibrations and ± 0.01° for the 120° nominal cone angle calibrations [4]. The complex microform geometric features of the Rockwell diamond indenter—the profile deviations from the least squares radius, the cone flank straightness, the holder axis alignment error and the surface finish roughness—can also be calibrated [4].

3. CALIBRATION RESULTS

The NIST microform calibration system was established in 1994. In 1995, a group of 11 Rockwell diamond indenters were calibrated by this system and yielded consistent geometric parameters. When these indenters were tested at the NIST standard Rockwell hardness testing machine, they also yielded consistent hardness performance [3]. The No. 3581 indenter from this group was selected as one of the NIST master standard Rockwell diamond indenters for the calibration of approximately 300 standard reference material (SRM) standard HRC blocks for establishment of the HRC scale in the United States [1, 5].

4. CALIBRATION REPRODUCIBILITY TESTS

The calibration reproducibility of the NIST microform calibration system and the long term stability of the NIST master standard Rockwell diamond indenters are both important for maintaining the established HRC scale in the U.S. For that reason, the No. 3581 indenter was re-calibrated in 1997, 2005 and 2007. The results from all three re-calibration runs, as well as the original calibration results from 1995, agree very well [6]. These results demonstrate both a high stability for the geometric parameters of the NIST master Rockwell indenter and high reproducibility for the microform calibration system.

After more than fifteen years of service, the old stylus instrument used for the microform calibration system was recently replaced by a new one. Meanwhile, some hardware and software have also been upgraded. In order to test the measurement reproducibility, another NIST master Rockwell indenter, No. 101, has been recently calibrated by the new instrument and compared with the old results calibrated by two other stylus instruments.

NIST 101 master Rockwell diamond indenter is used for the calibration of about 200 NIST standard reference material (SRM) Rockwell hardness blocks of HR15N and HR30N scales [7, 8]. Table 1 shows the calibration results for No. 101 master indenter calibrated in 1996, 2007 and 2009 using three different stylus instruments. The 1996 calibration used the original stylus instrument of the microform calibration system. The 2007 calibration used a demo instrument of the same type as the new instrument. The 2009 calibration uses the new stylus instrument, after the No. 101 indenter had been used for the calibrations of SRM Rockwell hardness blocks of HR15N and HR30N scales with about 2000 indentations. In Table 1, the last two calibration results dated on 4/07/2009 are from the same set of calibration data using the new instrument, but with different window sizes for data analyses. We will discuss it in next section. Uncertainties shown in Table 1 are reported for k = 2.

From Table 1, it can be seen that three calibrations for No. 101 indenter using three different instruments in 1996, 2007 and 2009 have very good agreement. For example, the mean spherical tip radius was calibrated to be 196.83 µm ± 0.70 µm in 1996, 196.76 µm ± 0.72 µm in 2007, and 197.09 µm ± 0.64 µm in 2007. The mean cone angle was calibrated to be 119.967° ± 0.020° in 1996, 119.969° ± 0.021° in 2007, and 119.962° ± 0.016° in 2009. The profile deviations from the least squares radii: the maximum peak height Pp and maximum valley depth Pv, and the cone flank straightness Pt also show good agreement (see Table 1). These results once again demonstrate the stability of the NIST master Rockwell diamond indenter and the long term reproducibility of the NIST microform calibration system. Both are important issues for maintaining the long term stability of the established national HRC scale.

5. WINDOW SIZES FOR THE CALIBRATION OF ROCKWELL INDENTERS

For the calibration of Rockwell diamond indenters, three window sizes must be previously specified for the data analyses to calculate the least squares radius and its profile deviations in the center and the cone angle and cone flank straightness in the left and right. For an ideally shaped Rockwell indenter as specified in the ASTM E18-07 and ISO 6508-3:2005(E) standards [7, 8], i.e., 200 µm tip radius blending with a 120° cone angle in a true tangential manner, the window sizes must be ± 100 µm for the tip radius calibration; and ± (100 to at least 446.4) µm along the x-axis for the left and right contributions to the cone angle calibration. The foregoing specification is calculated from the requirement of a "minimum length of 0.4 mm" along the sloping surface for the cone angle calibration as specified in ASTM and ISO standard [7, 8]. In practice, we round off the window to ± (100 to 450) µm (see Figure 1).

For the calibration of NIST master standard Rockwell indenters, we use a window size of ± 100 µm for the tip radius and profile deviation calibration and ± (100 to 450) µm for the left and right side of cone angle and flank straightness calibration. It works well for our standard Rockwell indenters with a shape close to the ideal shape. However, the Rockwell hardness test method standards of ASTM-International (ASTM) and the International Organization for Standardization (ISO) specify ranges of acceptable tolerances on the tip radius and cone angle [7, 8]. Depending on the actual radii of the indenters within these tolerances, the window sizes can vary by up to approximately ± 4 µm. An additional consideration is that industrial Rockwell indenters deviate slightly from the ideal shape in the transition area between the radial tip surface and the linear cone surface. Perhaps in light of these deviations, both the ASTM and ISO standards [7, 8] specify that the straightness of the cone flank is measured "adjacent to the blend" and so leaves some flexibility about the choice of position of the windows on the flanks.

Besides the window size of ± (100 to 450) µm we used for the calibration of our master standard indenters, we have recently tested the effect of different window positions for the calibration of cone angle and cone flank straightness of the industrial standard indenters. We tested several indenters using ± (105 to 450) µm and ± (110 to 450) µm window sizes for data analyses of the cone angle and cone flank straightness, and compared the results with those using the ± (100 to 450) µm window size.

In general, by narrowing the window size from ± (100 to 450) µm to ± (105 to 450) µm or ± (110 to 450) µm, the straightness error of cone flank is reduced. It also decreases the value of the cone angle, but not significantly. If the shape of the calibrated indenter is close to the ideal shape, the calibration differences would be small between the three different window sizes. As an example, Table 1 shows two sets of data analyses for the same set of calibration data of the No. 101 indenter recently calibrated on 4/07/2009, in which different window sizes of ± (100 to 450) µm and ± (105 to 450) µm are used for cone angle and cone flank straightness data analyses. It can be seen that the mean cone angle was calibrated as 119.962° ± 0.016° using ± (100 to 450) µm window; and 119.955° ± 0.016° using ± (105 to 450) µm window. The cone flank straightness Pt was calculated to be 0.51 µm using the ± (100 to 450) µm window; and 0.43 µm using ± (105 to 450) µm window (note: this is the largest of the 18 values calculated left and right from the nine sections). However, if the shape of the calibrated Rockwell indenter is near the limits of the tolerances, then the measurement differences caused by varying the window sizes could be significant, especially for the calibration of the cone flank straightness in an assessment of indenter quality.

6. SUMMARY

A new stylus instrument has been adapted to the calibration of the geometric parameters of Rockwell diamond indenters at NIST, replacing one in use since 1994. In order to test for the stability of the indenters and reproducibility for the geometric calibrations, the No. 101 NIST master Rockwell diamond indenter has been measured by the new stylus instrument and compared with the previous measurement results using two different stylus instruments. The three calibration results from 1996, 2007 and 2009 have shown very good agreement. The results demonstrate both the stability of the NIST master indenter and the long term reproducibility of the NIST calibration system.

For measurement of geometric parameters of Rockwell diamond indenters, the window positions and sizes must be specified in the data analysis. This is especially important for calculation of least squares radius and profile deviation in the center and cone flank straightness in the left and right. Furthermore, the blend area of the tip radius and cone angle must be accounted for when choosing window positions.

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Table 1. Calibration results for NIST master standard Rockwell indenter No. 101 in 1996, 2007 and 2009 using three different stylus instruments. Two sets of results in 2009 are from the same calibration data using the same window size of ± 100 µm for the calibration of tip radius and profile deviations, but using different window sizes of ± (100 to 450) µm and ± (105 to 450) µm for calibration of the cone angle and cone flank straightness. Uncertainties are reported for coverage factor, k = 2. Note: Parameters 1b, 2b, 3, and 4b are discussed in [4, 8, 9].

Microform Geometry Parameters	**Tolerances**	**Calibration Results**			
	Calibration Grade **Specified in ISO 6508-3:2005(E)**	**4/05/1996** **Window size: ± 100 µm and ± (100 to 450) µm**	**11/02/2007** **Window size: ± 100 µm and ± (100 to 450) µm**	**4/07/2009** **Window size: ± 100 µm and ± (100 to 450) µm**	**4/07/2009** **Window size: ± 100 µm and ± (105 to 450) µm**
1. Spherical Radius					
1a. Mean	200 µm ± 5 µm	(196.83 ± 0.70) µm	(196.76 ± 0.72) µm	(197.09 ± 0.64) µm	(197.09 ± 0.64) µm
1b. Maximum Variation	200 µm ± 7 µm	Max. = 198.25 µm	Max. = 198.15 µm	Max. = 198.15 µm	Max. = 198.15 µm
		Min. = 195.53 µm	Min. = 195.55 µm	Min. = 195.95 µm	Min. = 195.95 µm
1c. Profile Deviation	< 2 µm	Pp = 0.38 µm	Pp = 0.38 µm	Pp = 0.39 µm	Pp = 0.39 µm
		Pv = 0.34 µm	Pv = 0.31 µm	Pv = 0.31 µm	Pv = 0.31 µm
2. Cone Angle					
2a. Mean	120° ± 0.1°	119.967° ± 0.020°	119.969° ± 0.021°	119.962° ± 0.016°	119.955° ± 0.016°
2b. Maximum Variation	120° ± 0.17°	Max. = 120.005°	Max. = 120.014°	Max. = 119.990°	Max. = 119.985°
		Min. = 119.925°	Min. = 119.936°	Min. = 119.930°	Min. = 119.925°
2c. Cone Flank Straightness	< 0.5 µm	Pt = 0.46 µm	Pt = 0.48 µm	Pt = 0.51 µm	Pt = 0.43 µm
3. Holder Axis Alignment	0.3°	0.092°	---	---	---
4. Surface Finish					
4a. Roughness Mean	---	0.0043 µm	Ra = 0.0017 µm	Ra = 0.0021 µm	Ra = 0.0022 µm
4b. Max. Surface Roughness	---	0.0051 µm	Ra = 0.0020 µm	Ra = 0.0024 µm	Ra = 0.0022 µm