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GEOMETRICAL MEASUREMENTS OF NIST SRM ROCKWELL HARDNESS DIAMOND INDENTERS

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Abstract – The geometric parameters of 28 candidate Standard Reference Material (SRM) Rockwell hardness diamond indenters are calibrated by an automated system recently developed at the National Institute of Standards and Technology (NIST). The calibration results show that the cone flank straightness is a key issue for the calibrated indenters to be qualified for the geometrical specifications of NIST SRM diamond indenters. The effect of selecting different window sizes and locations for the evaluation of cone flank straightness is discussed.

Keywords: Rockwell C hardness, Rockwell diamond indenter, standard reference material

1. NIST SRM ROCKWELL HARDNESS DIAMOND INDENTER PROJECT

The geometric error of a Rockwell hardness diamond indenter is a major contributor to the measurement uncertainty of Rockwell C hardness (HRC) tests [1]. As a result, the geometric calibration of the diamond indenters is a key issue for HRC tests; and the development of Standard Reference Material (SRM) diamond indenters is an important step towards Rockwell hardness standardization in the United States. In 1994, NIST developed a microform calibration system based on a stylus instrument for the geometrical calibration of the diamond indenters [2]. This system has demonstrated high measurement reproducibility and low calibration uncertainty for the geometrical calibration of Rockwell hardness diamond indenters [3]. In 2009, an automated calibration system was established at NIST, which replaced the older system that relied on manual operation. The automated system has demonstrated the same calibration reproducibility and accuracy as the older system [3], but reduced the calibration time from hours to 20 minutes. It enables NIST to provide more efficient geometrical calibration services of Rockwell hardness diamond indenters for US and international customers.

A NIST SRM diamond indenter project is currently in progress. In 2009, the geometrical parameters of 28 candidate SRM indenters were calibrated by the automated system. It was found that the cone flank straightness is a key issue for the calibrated indenters to be qualified as NIST SRM indenters. In this paper, the calibration results are

analysed, and the effect of selecting different window sizes and locations for the evaluation of cone flank straightness is discussed.

2. TECHNICAL SPECIFICATIONS

The Rockwell hardness diamond indenter is a natural diamond stone, affixed into a metal holder, having a spherical conical shape of 120° cone angle blending with a 200 μm spherical tip radius. Geometrical parameters for the diamond indenters specified in ASTM E18 [4, 5] and ISO 6508 [6, 7] standards include:

- The mean tip radius, the maximum and minimum tip radius and form deviation;
- The mean cone angle, the maximum and minimum cone angle and cone flank straightness; and
- The holder axis alignment error.

The tolerance values for working and calibration grade diamond indenters specified in ASTM and ISO standards are summarized in Table 1. The working grade indenters are used for regular Rockwell hardness tests; the calibration grade indenters are used for calibration of reference HRC hardness blocks. For NIST SRM diamond indenters, most technical requirements are specified the same as those of the calibration grade indenters specified in the ASTM and ISO standards, except for the form deviation from the mean tip radius. Considering the significant effect of the spherical tip shape (sharp or flat) on hardness tests [8, 9], as well as the current industrial capability for manufacturing precise tip radius [8], we specified a tighter tolerance, 0.8 μm (see Table 1), for the form deviations from the mean radius, instead of 2 μm tolerance specified in ISO and ASTM standards. Furthermore, because the surface roughness of diamond indenters affects hardness tests [5, 6], we specified a tolerance for the surface roughness Ra of the SRM indenters, see Table 1. We also proposed tighter tolerances for the Reference Indenters—about half of the values specified for SRM indenter (see Table 1).

3. GEOMETRICAL CALIBRATIONS

The calibrations are performed at nine sections with 40° rotational increments on the Rockwell indenter. The expanded measurement uncertainties ($k = 2$) are $\pm 0.3 \mu\text{m}$ for the 200 μm tip radius calibrations and $\pm 0.01^\circ$ for the

Technical components:	Nominal values:	Tolerances specified in ASTM and ISO standards and the (numbers of qualified indenters) in 28 candidate SRM indenters			
		ISO 6508-2 ASTM E18 Working	ISO 6508-3 ASTM E18 Calibration	NIST SRM Indenter	Proposed Reference Indenter
1. Least squares radius and profile deviation:					
1.1. Mean radius:	200.00 μm Upper tolerance (μm) Lower tolerance (μm)	210 (28) 190 (28)	205 (27) 195 (27)	205 (27) 195 (27)	202.5 (26) 197.5 (22)
1.2. Max. radius:	Tolerance (μm)	215 (28)	207 (26)	207 (26)	203.5 (20)
Min. radius:	Tolerance (μm)	185 (28)	193 (28)	193 (28)	196.5 (21)
1.3. Profile deviations:					
Max. profile peak height Pp :	(μm)	2 (ASTM, 28)	2 (ASTM, 28)	0.8 (28)	0.4 (11)
Max. profile valley depth Pv :	(μm)	2 (ASTM, 28)	2 (ASTM, 28)	0.8 (28)	0.4 (17)
Max. Total profile height Pt :	(μm)	4 (ISO, 28)	2 (ISO, 28)		
2. Cone angle and cone flank straightness:					
2.1. Mean cone angle:	120.00° Upper tolerance ^(*) Lower tolerance ^(*)		120.1* (26) 119.9* (24)	120.1 (26) 119.9 (24)	120.05 (25) 119.95 (16)
2.2. Max. cone angle:	Tolerance ^(*)	120.35 (28)	120.17 (27)	120.17 (27)	120.08 (26)
Min. cone angle:	Tolerance ^(*)	119.65 (28)	119.83 (27)	119.83 (27)	119.92 (17)
2.4. Max. cone flank straightness Pt :	(μm)	2 (ISO, 28)	0.5 (ISO, 3) 2 (ASTM, 28)	0.5 (3)	0.3 (1)
3. Holder axis alignment error:	^(*)	0.5 (28)	0.3 (28)	0.3 (28)	0.15 (10)
4. Surface roughness:					
4.1. Mean Ra :	(μm)			0.005 (28)	0.003 (22)
4.2. Max. Ra :	(μm)			0.007 (28)	0.005 (28)
How many indenters among the 28 indenters are overall qualified?	%	28/28 100%	2/28 7%	2/28 7%	0/28 0

Table 1. Technical specifications for Rockwell hardness diamond indenters in ASTM E18, ISO 6508 standards [4-7], and for NIST SRM and the proposed Reference Indenters (*: Specified in ASTM E18-05 as the maximum and minimum value). Among the 28 calibrated indenters using the “adjacent” windows of $\pm 95 \mu\text{m}$ and $\pm (105 \text{ to } 455) \mu\text{m}$, the number of indenters that passed the specification for each grade of indenter is shown in the bracket.

120° cone angle calibrations [2]. The microform geometric features of the Rockwell diamond indenter and the surface roughness can also be calibrated.

For ideally shaped diamond indenters with 120° cone angle blending with a 200 μm spherical tip radius in a true tangential manner, the calibration windows can be selected as $\pm 100 \mu\text{m}$ for the tip radius calibration and $\pm (100 \text{ to } 450) \mu\text{m}$ for the left and right cone angle calibration [2, 3]. However, the blend point can vary position depending on the actual cone angle and tip radius. Industrial diamond indenters deviate slightly from the ideal shape, especially in the transition area between the radial tip surface and the linear cone surface (see Figure 1). Perhaps in light of these deviations, both the ASTM and ISO standards specify that the straightness of the cone flank is measured “adjacent to the blend” [4-7] which leaves some flexibility in the choice of the size and position of the windows on the flanks. We have tested different window sizes and positions for the calibration of diamond indenters.

First, we tested “adjacent” windows at $\pm 95 \mu\text{m}$ for the radius calibration and $\pm (105 \text{ to } 455) \mu\text{m}$ at left and right for the cone angle calibration. Table 1 shows the numbers (in the bracket) of qualified diamond indenters among the 28 calibrated indenters for each tolerance specification. Most test results were similar to those obtained using the standard windows of $\pm 100 \mu\text{m}$ and $\pm (100 \text{ to } 450)$. For most indenters, their geometrical parameters passed the technical specifications for ASTM and ISO calibration grade indenter,

as well as a NIST SRM indenter, except that only three indenters passed the technical specification of 0.5 μm for the cone flank straightness as specified in ISO 6508-3 [7]; two of them are qualified overall as NIST SRM indenters (see Table 1, bottom). When using the standard window $\pm (100 \text{ to } 450) \mu\text{m}$ for cone straightness calibration, none qualified the 0.5 μm certification.

4. THE EFFECT OF WINDOW SIZE AND POSITION

For a precise and repeatable calibration of diamond indenters, the window size and position must be previously defined. For the calibration of NIST master standard diamond indenters, we use the windows of $\pm 100 \mu\text{m}$ for tip radius and profile deviation calibration and $\pm (100 \text{ to } 450) \mu\text{m}$ for left and right side of cone angles and flank straightness calibration [2, 3]. It works well for our standard diamond indenters with a shape close to the ideal shape. Considering the large straightness error of the industrial indenters and that both ASTM and ISO standards specified the cone flank straightness is measured “adjacent to the blend” [4-7], we tested windows of $\pm 95 \mu\text{m}$ and $\pm (105 \text{ to } 455) \mu\text{m}$ as mentioned before; we have also tested a $\pm 100 \mu\text{m}$ window for tip radius calibration and $\pm (110 \text{ to } 460) \mu\text{m}$ windows for cone angle calibration. Most test results were similar to those obtained using the other two sets of windows, except that the qualified indenters for the 0.5 μm straightness specification increased from three to eight; and

four of them then qualified overall as NIST SRM indenters (see Table 2). However, excluding part of inside curvature on the cone profile will decrease the value of cone angle measurements. It can be seen in Figure 1: when the left and right windows are expanded to outside for cone angle calibrations, part of the inside profile curvature located at “A” and “B” would be excluded, that could reduce the cone flank straightness error. However, it might turn up of the inside part of the left and right least squares mean lines, that

would make the conical cone “sharper”, or decrease the cone angle. As a result, when the window position was expanded to $\pm (120 \text{ to } 470) \mu\text{m}$ for the cone angle evaluations, 20 of the indenters passed the $0.5 \mu\text{m}$ straightness specification, but only 12 of them qualified overall as SRM indenters (see Table 2). That is because for some indenters, the average and the minimum cone angle values were decreased below their minimum specifications of 119.9° for the mean value, and 119.83° for the minimum value.

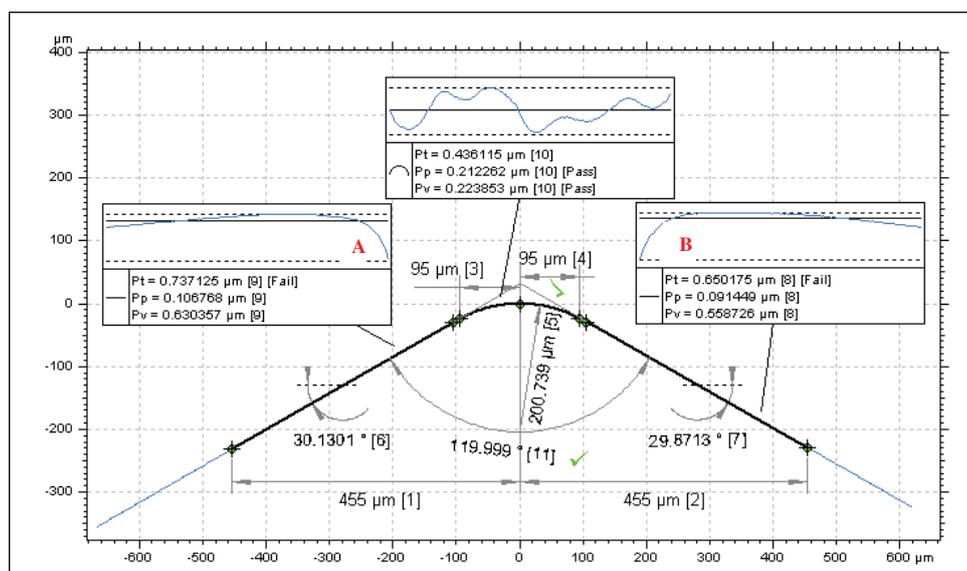


Figure 1. An indenter profile processed using the “adjacent” windows of $\pm 95 \mu\text{m}$ for the radius measurement and $\pm (105 \text{ to } 455) \mu\text{m}$ for the cone angle measurement. Note: The numbers in brackets represent entries in a spreadsheet for tracing measured results.

5. DISCUSSION

In the ASTM E18 standards [4, 5], the tolerance of form deviation from the mean radius (meaning either the maximum profile peak height Pp or the maximum profile valley depth Pv as defined in the ASME B46 standard [10]) is specified as $2 \mu\text{m}$ for both the working and calibration grade indenters. In the ISO standards [6, 7], the tolerance for the total profile height $Pt = Pp + Pv$ is specified as $4 \mu\text{m}$ for working grade indenters and $2 \mu\text{m}$ for calibration grade indenters (see Table 1). Both Rockwell hardness tests and Finite Element Analyses (FEA) simulation results have shown that the spherical tip shape (sharp or flat) of diamond indenters has a significant influence on hardness readings, and that the $2 \mu\text{m}$ tolerance for the form deviation is not a sufficiently tight control for the tip shape [8, 9]. On the other hand, from our experience of measuring diamond indenters obtained from different manufacturers in the world, we have found that the actual form deviations of most diamond indenters are well below $1 \mu\text{m}$. As a result, we specify a $0.8 \mu\text{m}$ tolerance for the maximum form deviations of NIST SRM Indenters, and $0.4 \mu\text{m}$ for the proposed Reference Indenters (see Table 1). The calibration results for 28 candidate SRM indenters have shown that all their maximum form deviations (Pp or Pv) are less than $0.8 \mu\text{m}$, with 11 indenters showing the maximum peak height (Pp) less than $0.4 \mu\text{m}$, and 17 indenters showing the maximum valley depth (Pv) less than $0.4 \mu\text{m}$ (see Table 1).

On the other hand, the tolerance for cone flank straightness (meaning the total profile height $Pt = Pp + Pv$ [10]) specified in ISO 6508-3 standard [7] is a very tight $0.5 \mu\text{m}$. It is difficult to produce a diamond indenter with a $100 \mu\text{m}$ tip radius blending with a 120° cone angle in a true tangential manner. As a result, the $0.5 \mu\text{m}$ tolerance for the cone flank straightness might be too tight for manufacturers. In our experience of measuring diamond indenters from different manufacturers, only a few indenters qualified the $0.5 \mu\text{m}$ straightness specification. When the standard windows $\pm (100 \text{ to } 450) \mu\text{m}$ are used for cone flank straightness calibration, none qualified under the $0.5 \mu\text{m}$ specification among the 28 calibrated indenters. Even using the “adjacent” windows of $\pm (105 \text{ to } 455) \mu\text{m}$, there are only three indenters among the 28 calibrated indenters that passed the $0.5 \mu\text{m}$ for cone flank straightness (see Table 1). If the window position is expanded to $\pm (120 \text{ to } 470) \mu\text{m}$ for cone angle calibrations, more indenters (20) qualify under the $0.5 \mu\text{m}$ straightness specification, but the values for cone angle might decrease significantly.

6. SUGGESTIONS

From the calibration results of 28 candidate SRM indenters, it can be seen that the cone flank straightness is a key issue for these indenters to be qualified for the geometrical specifications as NIST SRM diamond indenters. This is mostly because of the tight tolerance, $0.5 \mu\text{m}$,

Windows for straightness evaluation (μm)	Number of indenters qualified overall as grade of			
	ISO 6508-2	ISO 6508-3	NIST	NIST
	ASTM E18 Working	ASTM 18 Calibration	SRM Indenter	Standard Indenter
\pm (100 to 450)	28/28	0/28	0/28	0/28
\pm (105 to 455)	28/28	2/28	2/28	0/28
\pm (110 to 460)	28/28	4/28	4/28	0/28
\pm (120 to 470)	28/28	12/28	12/28	0/28

Table 2. Summary of the calibration results of 28 SRM indenters using four windows for cone angle straightness evaluation.

specified in ISO 6508-3 standard [7], as well as the ambiguous measurement windows specified in both ASTM and ISO standards [4-7] for evaluation of cone flank straightness. Some possible solutions are suggested as follows:

1. Expand window position: In order to specify an expand window position “adjacent to the blend” for the calibration of cone flank straightness without changing the measurement results of cone angles, it is suggested to use a standard window \pm (100 to 450) μm for the cone angle calibration and use expanded windows, for example \pm (110 to 450) μm or \pm (110 to 460) μm , for the cone flank straightness evaluation.

2. Use “form deviation” rather than “straightness” as a control parameter: Use the “form deviation” (maximum profile peak height Pp or maximum profile valley depth Pv [10]) as the control parameter for the cone flank form error, instead of using “straightness” (total profile height $Pt = Pp + Pv$ [10]). This change would significantly relax the tolerance for cone flank form error. For example, when the cone flank is evaluated within the windows of \pm (110 to 460) μm , if the 0.5 μm tolerance for “straightness” ($Pt = Pp + Pv$) is used for control of the form error, eight Rockwell indenters among the 28 calibrated indenters would qualify under this specification. If the 0.5 μm “form deviation” (Pp or Pv) is specified instead of the “straightness” ($Pt = Pp + Pv$), the number of qualified Rockwell indenters would be increased from eight to 20.

3. Use the “mean and maximum” of straightness: Use 0.5 μm as the “mean value” of the straightness measured at nine sections (or other section numbers) combined with the use of a 0.8 μm “maximum” straightness value as the control parameters for the cone flank straightness, instead of the currently used 0.5 μm “maximum” straightness. This could also significantly relax the tolerance for cone flank form error. For example, when the cone flank is evaluated in the windows of \pm (110 to 460) μm , if the 0.5 μm “mean” straightness and the 0.8 μm “maximum” straightness are used for control of the form error, instead of using the 0.5 μm “maximum” straightness, the number of qualified SRM indenter numbers would be increased from eight to 21.

It is important that the geometrically qualified diamond indenters, i.e. the indenters passed the direct verification [4-7], must under hardness performance tests, i.e., indirect verification [4-7]. For NIST SRM diamond indenters, NIST master diamond indenter and SRM hardness blocks are used as the comparison reference for the indirect verifications.

7. SUMMARY

The geometrical measurements of 28 NIST candidate SRM diamond indenters show that, the cone flank straightness is a key quality issue. This is a consequence of the technical specification of the current Rockwell hardness standards [4-7], in which a very large tolerance, 2 μm , is specified for the form deviations of the tip radius, while a very tight tolerance, 0.5 μm , is specified in ISO 6508-3 [7] for the cone flank straightness ($Pt = Pp + Pv$).

Industrial Rockwell indenters deviate slightly from the ideal shape, especially 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 specify that the straightness of the cone flank is measured “adjacent to the blend” [4-7] and so it leaves some flexibility about the choice of the size and position of the windows on the flanks. However, for any precise and repeatable calibration of Rockwell indenters, the window size and position must be previously defined. This is extremely important for the coming CIPM key comparisons for international Rockwell hardness tests of the Working Group on Hardness (WGH) in the framework of the Consultative Committee of Weights and Measures (CCWM) of CIPM.

REFERENCES

- [1] Liggett, W., Low, S., Pitchure, D., and Song, J., “Capability in Rockwell C scale hardness,” *J. Res. NIST*, **105**(4), 511-533, 2000.
- [2] Song, J., Rudder, F., Vorburger, T., and Smith, J., “Microform calibration uncertainties of Rockwell diamond indenters,” *J. Res. NIST*, **100**(5), 543-561, 1995.
- [3] Song, J., Low, S., Zheng, A., “Geometric measurement comparisons for Rockwell diamond indenters” in the proceedings of the IMEKO XIX World Congress, Lisbon, Portugal, 2009.
- [4] ASTM E18-07 *Standard Test Method for Rockwell Hardness of Metallic Materials*, ASTM, Philadelphia, 2007.
- [5] ASTM E18-05, *Standard Test Method for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials*, ASTM, Philadelphia, 2005.
- [6] ISO 6508-2:2005(E) *Metallic Materials – Rockwell hardness test – Part 2: Verification and calibration of testing machines (scales A, B, C, D, E, F, G, H, K, N, T)*, ISO, Geneva, 2005.
- [7] ISO 6508-3:2005(E) *Metallic Materials – Rockwell hardness test – Part 3: Calibration of reference blocks (scales A, B, C, D, E, F, G, H, K, N, T)*, ISO, Geneva, 2005.
- [8] Song, J., Low, S., and Ma, L., “Tolerancing form deviations for NIST standard reference material (SRM) 2809 Rockwell diamond indenters,” in Proc. International Symposium on Advances in Hardness Measurement (HARDMEKO 2007), NRLM, Tsukuba, Japan, 97-102, 2007.
- [9] Ma, L., Low, S., Zhou, J., Song, J., and DeWit, R., “Simulation and prediction of hardness performance of Rockwell diamond indenters using finite element analysis,” *Journal of Testing and Evaluation*, **30**(4), ASTM International, PA, 265-273, 2002.
- [10] ASME B46.1-2002, *Surface Texture (Surface Roughness Waviness and Lay)*, America Society of Mechanical Engineers (ASME), New York, 2003.