XXI IMEKO World Congress "Measurement in Research and Industry"
August 30 – September 4, 2015, Prague, Czech Republic

DEVELOPING NIST ROCKWELL HARDNESS DIAMOND INDENTER SRMS BASED ON ISO 6508-3:2015 STANDARD

John Song, *Samuel Low*, *Thomas B. Renegar* and *Li Ma*
National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899, USA
Emails: song@nist.gov; samuel.low@nist.gov; thomas.renegar@nist.gov; li.ma@nist.gov

Abstract – The ISO 6508-3:2005 standard specified a tight tolerance for the cone flank straightness of Rockwell diamond indenters (0.5 µm for maximum). In the recent issued ISO 6508-3:2015 standard, significant changes on the microform geometrical specification of Rockwell diamond indenters and calibration conditions have been made: the tolerance range for cone flank straightness has been relaxed from "0.5 µm for maximum" to "0.5 µm for average and 0.7 µm for maximum"; and the window size and location for calibration of the Rockwell diamond indenters have been specified. Based on the technical specification of ISO 6508-3:2015, NIST researchers are working with diamond manufacturers to develop the Standard Reference Material (SRM) Rockwell hardness diamond indenters to support U.S. Rockwell hardness standardization.

Keywords: Rockwell C hardness, HRC, microform calibration, diamond indenters, ISO 6508-3:2015.

1. BACKGROND

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 [1]. A Rockwell hardness scale is established by the performance of a standard Rockwell diamond indenter (for the HRC, HRD, HRA, HR45N, HR30N and HR15N scales) using a standard testing machine and a standardized testing cycle [1]. In the uncertainty budget of Rockwell hardness tests, the geometric errors of the diamond indenter are a major contributor [2]. International comparisons showed that, when a "common indenter" was used at different national laboratories for hardness tests, the variation range is much less than that of using different national indenters [3].

The National Institute of Standards and Technology (NIST) is developing Rockwell hardness diamond indenter standard reference materials (SRMs) to support Rockwell hardness standardization and measurements in the U.S. One of the key steps is the microform calibration of geometrical parameters of the SRM indenters. Since the former ISO standard published in 1986 (ISO 715:1986) and revised in 2005 (ISO 6508-3:2005) [4] specified a tight tolerance for the cone flank straightness of Rockwell diamond indenters (0.5 µm for maximum), which was beyond the current production capability of the diamond manufacturers, few indenters calibrated at NIST could meet this tight specification. Furthermore, the window size and location for calibration of the Rockwell indenters were not specified in the former ISO standard [4], which made it difficult for accurate calibrations.

In the recently issued ISO 6508-3:2015 standard entitled "*Metallic materials — Rockwell hardness test — Part 3: Calibration of reference blocks*" [5], significant changes on the microform geometrical specification of Rockwell diamond indenters and calibration conditions have been made based on NIST's research and suggestions [6 to 8]: the tolerance range for cone flank straightness has been relaxed from "0.5 µm for maximum" [4] to "0.5 µm for average and 0.7 µm for maximum" [5]; and the window size and location for calibration of the Rockwell diamond indenters have been specified [5], which have made it possible for accurate calibration of microform geometry of Rockwell diamond indenters.

In this paper, we describe the geometrical specification of the SRM indenters and the capability of the NIST microform calibration system in Sections 2 and 3; we describe the initial calibration results and suggestions in Section 4. Finally, we discuss our plan to develop standard Rockwell diamond indenter SRMs based on the newly issued ISO 6508-3:2015 standard.

2. GEOMETRICAL SPECIFICTIONS FOR NIST ROCKWLL DIAMOND INDENTER SRMS

The Rockwell hardness diamond indenter is made of a natural diamond stone, affixed into a metal holder; the diamond has a spherical conical shape of 120° cone angle blending with a 200 µm spherical tip radius (Fig. 1). Geometrical parameters for the diamond indenters specified in ISO 6508-3:2005 and ISO 6508-3:2015 standards include [4, 5]:

- 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.

Two grades of Rockwell diamond indenters, working grade and calibration grade, are specified in the ISO standards. The working grade indenters are used for regular Rockwell hardness tests; the calibration grade indenters, specified with tight tolerances for both the geometrical parameters and the hardness performances, are primarily used for calibration of reference hardness blocks [4, 5].

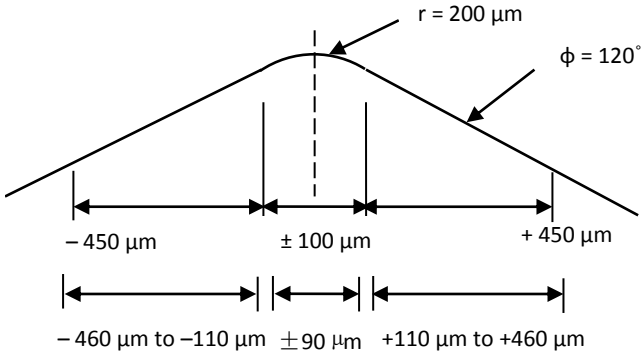

Figure 1. Three nominal windows for data fitting of tip radius and profile deviations at ± 100 µm of the center, and for cone angle and cone flank straightness fitting at ± (100 to 450) µm of the left and right. The actual windows for NIST calibration of SRM Rockwell diamond indenters are ± 90 µm for tip radius calibrations and ± (110 to 460) µm for cone angle calibrations.

For the technical specifications of NIST Rockwell diamond indenter SRMs, the bottom line is that these specifications must meet the highest level published in the international test method. On the other hand, in case of ensuring the high quality of the SRM Rockwell diamond indenters, some technical specifications for SRM indenters could be even higher, but absolutely should not be lower, than those specified in the international test method.

Most technical requirements of the NIST Rockwell diamond indenter SRMs are specified the same as those of the calibration grade Rockwell diamond indenters specified in the ISO standards [4, 5], 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 [6 to 8], as well as the current industrial capability for manufacturing the precise shape of tip radius, a tighter tolerance, 0.8 µm for the maximum profile height deviation *P*p and the maximum profile valley depth *P*v, was specified for the NIST SRM indenters (Table 1) [9]. In the ISO ISO 6508-3:2005 and ISO 6508-3:2015 standards, 2 µm tolerance of total profile peak height *P*t is specified [4, 5]. Furthermore, because the surface roughness of diamond indenters affects hardness tests [4, 5], a tolerance for the surface finish roughness *R*a was specified for the proposed Rockwell diamond indenter SRMs [9]. The technical specifications in ISO 6508-3:2005 and ISO 6508-3:2015 standards for the calibration grade Rockwell hardness diamond indenters and the proposed NIST SRM indenters, and NIST calibration uncertainties are shown in Table 1.

Table 1. Technical specifications in ISO 6508 standards for the calibration grade Rockwell hardness diamond indenters and the proposed NIST SRM indenters, and NIST calibration uncertainties

	Tolerances Specified in ISO Standards and NIST SRM Indenters			
Technical components:	ISO 6508-3: 2005 Calibration	ISO 6508-3: 2015 Calibration	NIST SRM Indenter	NIST Calibration Uncertainty
1. Least squares radius and profile deviation:				
1.1. Mean radius (µm):	200 ± 5	200 ± 5	200 ± 5	0.3
1.2. Max. and Min. radius (µm):	200 ± 7.5	200 ± 7.5	200 ± 7.5	0.3
1.3. Profile deviations:				
Max. profile peak height Pp (µm):			0.8	0.1
Max. profile valley depth Pv (µm):			0.8	0.1
Max. total profile height Pt (µm):	2	2		
2. Cone angle and cone flank straightness:				
2.1. Mean cone angle (°) :	120° ± 0.1°	120° ± 0.1 °	120° ± 0.1°	0.01°
2.2. Max. and Min cone angle (°):	120° ± 0.17°	120° ± 0.17°	120° ± 0.17°	0.01°
2.4. Max. cone flank straightness Pt (µm):	0.5	0.7	0.7	0.05
Mean cone flank straightness (µm):		0.5	0.5	0.05
3. Holder axis alignment error (°):	0.3°	0.3°	0.3°	0.025°
4. Surface roughness:				
4.1. Mean Ra (µm):			0.005	0.001
4.2. Max. Ra (µm):			0.007	0.001

3. CAPABILITY OF NIST GEOMETRICAL CALIBRATION FOR ROCKWELL DIAMOND INDENTERS

The geometric error of a Rockwell hardness diamond indenter is a major contributor to the measurement uncertainty of Rockwell hardness tests [2]. The development of Rockwell diamond indenter SRMs with accurately calibrated geometrical parameters is an important step towards Rockwell hardness standardization. In 1994, NIST developed a microform calibration system based on a stylus instrument, combined with a set of calibration and check standards and a set of calibration and uncertainty procedures, for the geometrical calibration of the diamond indenters [3]. This system has demonstrated high measurement reproducibility and low calibration uncertainty for the geometrical calibration of Rockwell hardness diamond indenters [3, 9, 10]. The expanded measurement uncertainties (k = 2) for the calibration of Rockwell diamond indenters are ± 0.3 μm for the indenter tip radii calibrations and ± 0.01° for the cone angle calibrations [3]. The complex microform geometric features of the Rockwell diamond indenter, including 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 [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 [9], 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 U.S. and international customers.

4. INITIAL CALIBRATION RESULTS AND DISCUSSION

The initial geometrical calibration for 28 candidate SRM diamond indenters showed that the cone flank straightness was a key quality issue [9]. It was found that the 0.5 μm tolerance of cone flank straightness specified in the former ISO standard [4] might be too tight to fit in the current production capability of the diamond manufacturers. As a result, most candidate Rockwell diamond indenters calibrated at NIST could meet almost all the technical requirements, including the ± 0.8 μm reduced tolerance for the form deviation from the tip radius, except for the 0.5 μm tolerance of the cone flank straightness specified in the former ISO standard [4]. This was a consequence of the technical specification of the former ISO 6508-3:2005 standards [4] in which a tight tolerance, 0.5 μm, was specified for the cone flank straightness; as well as the calibrations were performed under the nominal window sizes of ± 100 μm and ± (100 to 450) μm (Fig. 1).

The microform geometries of 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, the ISO standards specify that the straightness of the cone flank is measured "adjacent to the blend" [4, 5] 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 Key Comparisons (KC) and pilot study for international Rockwell hardness tests (CCM.H-K3) organized by the Working Group on Hardness (WGH) in the framework of the Consultative Committee for Mass and Related Quantities (CCM) of the International Committee for Weights and Measures (CIPM).

5. DEVELOPING ROCKWELL DIAMOND INDENTER SRMS BASED ON ISO 6508-3:2015 STANDARD

Since the late 1990's, based on the microform calibrations and finite element simulations [6 to 8], NIST researchers suggested relaxing the tolerance range for cone flank straightness, and specifying the window size and location for accurate calibration of the Rockwell diamond indenters.

In the recently issued ISO/6508-3:2015 standard [5], significant changes on the microform geometrical specification of Rockwell diamond indenters and calibration conditions have been made based on these suggestions: the tolerance range for cone flank straightness has been relaxed from "0.5 μm for maximum" to "0.5 μm for average and 0.7 μm for maximum". Meanwhile, the window size and location for calibration of the Rockwell diamond indenters have been specified [5], which have made it possible for accurate calibration of microform geometry of Rockwell diamond indenters.

For the window size and locations for calibration of the Rockwell diamond indenters, the ISO 6508-3:2015 specification states that [5]:

"The surfaces of the cone and the spherical tip shall blend in a smooth tangential manner. The location where the spherical tip and the cone of the diamond blend together will vary depending on the values of the tip radius and cone angle. Ideally for a perfect indenter geometry, the blend point is located at 100 μm from the indenter axis measured along a line normal to the indenter axis. To avoid including the blend area in the measurement of the tip radius and cone angle, the portion of the diamond surface between 90 μm and 110 μm should be ignored."

The ISO 6508-3:2015 standard also specifies a minimum length of 0.4 mm for calibration of cone angle and cone flank straightness [5]:

"The mean deviation from straightness of the generatrix of the diamond cone, adjacent to the blend, shall not exceed 0,0005 mm over a minimum length of 0,4 mm. In each measured section, the deviation shall not exceed 0,0007 mm."

The nominal and the actual window size and location for microform calibration of Rockwell diamond indenters are shown in Fig. 1. Considering the ISO 6508-3:2015 specification of a minimum length of 0.4 mm for calibration of the cone flank straightness [5], the actual window size and position for calibration of the SRM indenters are specified as ± 90 µm for tip radius and form deviation calibrations and ± (110 to 460) µm, rather than ± (110 to 450) µm, for cone angle and cone flank straightness calibrations (Fig. 1). Since the ± (110 to 450) µm window will result in a calibration length for cone flank straightness less than 0.4 mm.

NIST researchers are currently working with diamond manufacturers to develop the Rockwell hardness diamond indenter SRMs based on the new technical specifications included within the ISO 6508-3:2015 revision of the standard.

ACKNOWLEDGMENTS

The authors are grateful to A. Zhang and P. Gu for their measurements of the candidate NIST SRM diamond indenters.

REFERENCES

[1] S. Low, *Rockwell Hardness Measurement of Metallic Materials,* NIST Special Publication 960-5, National Institute of Standards and Technology (2001).

[2] W. Liggett, S. Low, D. Pitchure, and J. Song, "Capability in Rockwell C scale hardness," *J. Res. NIST*, 105(4), 511-533 (2000).

[3] J. Song, S. Low, D. Pitchure, A. Germak, S, DeSogus, T. Polzin, H. Yang, H. Ishida, and G. Barbato, "Establishing a world-wide unified Rockwell hardness scale with metrological traceability," *Metrologia,* 34(4), BIPM, Paris, 331-342 (1997).

[4] 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.

[5] ISO 6508-3:2015 (E) "*Metallic materials — Rockwell hardness test — Part 3: Calibration of reference blocks*," ISO Geneva, 2015.

[6] J. Song, S. Low, L. Ma, "Form error and hardness performance of Rockwell diamond indenters," in *Proc. 16th IMEKO World Congress*, p.I-95, IMEKO, Vienna, Austria, 2000.

[7] L. Ma, S. Low, J. Zhou, J. Song, and R. DeWit, "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.

[8] J. Song, S. Low, and L. Ma, "Tolerancing form deviations for NIST standard reference material (SRM) Rockwell diamond indenters," in *Proc. International Symposium on Advances in Hardness Measurement* (HARDMEKO 2007), NRLM, Tsukuba, Japan, 97-102, 2007.

[9] J. Song, S. Low, A. Zheng, and P. Gu, "Geometrical measurements of NIST SRM Rockwell hardness diamond indenters," IMEKO 2010 TC3, TC5 and TC22 Conferences, Pattaya, Thailand, 2010.

[10] J. Song, S. Low, A. Zheng, "Calibration reproducibility test for NIST No 3581 standard Rockwell diamond indenter," in *Proc. XVIII IMEKO World Congress*, p.TC5-2, IMEKO, Rio de Janeiro, Brazil, 2006.