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TOLERANCING FORM DEVIATIONS FOR NIST STANDARD REFERENCE MATERIAL (SRM) 2809 ROCKWELL DIAMOND INDENTERS

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Abstract – The National Institute of Standards and Technology (NIST) is developing Standard Reference Material (SRM) 2809* Rockwell Diamond Indenter to support Rockwell hardness standardization. Most tolerances of the SRM indenters are adopted from those of the calibration grade Rockwell indenters specified in ASTM and ISO standards except the form deviations from tip radius. Based on the historical data of geometrical measurements and hardness tests at NIST, and the results of FEA (Finite Element Analyses) simulations, a tight tolerance, ± 0.5 µm, is specified for the form deviations of the SRM indenters.

Keywords: Rockwell hardness, diamond indenter, form deviation

1. INTRODUCTION

Rockwell hardness is the most widely used mechanical testing method for metal products. 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 for Rockwell diamond indenters have made it possible to establish a worldwide unified Rockwell hardness scale with metrological traceability [2]. This includes establishment of both the reference standards (standard machine and diamond indenter) traceable to SI units of force, time and length, and the reference testing conditions (a standardized common testing cycle) based on an international agreement.

The Rockwell diamond indenter is a diamond cone with 120E cone angle blended in a tangential manner with a spherical tip of 200 µm radius. Geometric parameters of the Rockwell diamond indenter include tip radius and form deviations, cone angle and cone flank straightness, and holder alignment error. In the uncertainty budget of Rockwell hardness tests, geometric errors of the diamond indenter play a dominant part [3]. The National Institute of Standards and Technology (NIST) is developing Standard Reference Material (SRM) 2809 Rockwell indenter to support Rockwell hardness standardization. These SRM indenters are characterized by geometric and hardness performance uniformity, measurement traceability, stability and interchangebility [4]. Most tolerances for the NIST SRM indenters are adapted from those of the calibration grade Rockwell indenters specified in ASTM (ASTM-International) and ISO (International Organization of Standardization) standards [5-7]. However, based on the results of geometrical measurements, hardness tests and FEA (Finite Element Analyses) simulations, we believe that the form deviations from the tip radius specified in both ASTM standard (± 2 µm [5]) and ISO standards (4 µm [6] and 2 µm [7]) are too large to meet the performance uniformity requirements, especially for the calibration grade Rockwell indenter. We have specified ± 0.5 µm tolerance for the form deviations of NIST SRM Rockwell indenters.

2. DEVELOPMENT HISTORY OF THE TOLERANCE OF ROCKWELL DIAMOND INDENTERS

There are two grades of Rockwell diamond indenters specified in ASTM and ISO standards [5-7]. The working grade indenters specified in ASTM E18-05 [5] and ISO 6508-2 [6] are used for ordinary hardness tests. The calibration grade indenters specified in ASTM E18-05 [5] and ISO 6508-3 [7] are used for calibrations of Rockwell hardness blocks. The geometric tolerances and hardness performance uniformity for the two grades of diamond indenters are shown in Table 1.

Historically, optical projection was the only method for inspection of the spherical tip of Rockwell diamond indenters. In the old version of ASTM E18-74/79/84 [8] and ISO R716-1968 [9] standards, a ± 2 µm tolerance bend was specified for a comprehensive inspection of the spherical tip of Rockwell diamond indenters using a 500× optical projector. From Fig. 1 [8, 9], it can be seen that this inspection was mainly used for control of the tip radius. For example, profile AB with a small tip radius and profile CD

* Certain commercial equipment, instruments, or materials are identified in this paper to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

TABLE 1. Technical Specifications for Working and Calibration Grade, and NIST SRM 2809 Rockwell Diamond Indenters

Microform Geometry and Performance Uniformity Requirements	Tolerance			NIST Expanded Measurement Uncertainty (k = 2)
	Working Grade (ASTM E18-05 [5], ISO/6508-2 [6])	**Calibration Grade (ASTM E18-05 [5], ISO/6508-3 [7])**	**NIST SRM 2809 Rockwell Diamond Indenter**	
1. Spherical Radius				
1a. Least Squares Mean	200 ± 10 µm	200 ± 5 µm	200 ± 5 µm	0.3 µm
1b. Maximum Variation	200 ± 15 µm	200 ± 7 µm	200 ± 7 µm	
1c. Profile Deviation	± 2 µm(5), 4 µm(6)	± 2 µm(5), 2 µm(7)	± 0.5 µm	
2. Cone Angle				
2a. Least Squares Mean	---	120 ± 0.1	120 ± 0.1	0.01
2b. Maximum Variation	120 ± 0.35	120 ± 0.17 (7)	120 ± 0.17	
2c. Cone Flank Straightness	< 2 µm(7)	< 0.5 µm(7)	< 0.5 µm	
3. Holder Axis Alignment	± 0.5	± 0.3	± 0.3	0.05
4. Surface Finish				
4a. Surface Roughness Mean	---	---	R_a < 0.005 µm	
4b. Max Surface Roughness	---	---	R_a*max* < 0.006 µm	
Performance Uniformity Requirements	± (0.5 - 1) HR(6) ± 0.8 HR(7) with respect to a standard indenter	± 0.4 HR(7) with respect to a standard indenter	± 0.4 HR with respect to NIST Standard Rockwell Diamond Indenter	

with a large tip radius (see Fig. 1b) are both qualified, because both of them are located within the ± 2 µm tolerance band. Since 1980's, different techniques and instruments have been developed for geometrical measurements of Rockwell diamond indenters. Those include contact stylus instrument [10] and non-contact optical instruments [11, 12]. Meanwhile, in the recent version of ASTM E18-5 [5] and ISO 6508-2/3 standards [6, 7], tolerances are individually specified for direct verification of each microform geometric parameters. Those include tip radius and form deviation, cone angle and cone flank straightness, and holder axis alignment (see Table 1). For the calibration grade Rockwell indenters, tolerance of the mean cone angle was specified as 120° ± 0.1°, with a tolerance of the cone flank straightness of 0.5 µm [5, 7]; tolerance of the mean tip radius was specified as 200 µm ± 5 µm [5, 7]. However, in the ASTM E18-5 standard [5], the form deviation from the tip radius was specified as ± 2 µm, the same as the old ASTM E18 [8] and ISO standard [9] specified mainly for inspection of the tip radius. In the recent ISO 6508-2/3 standards [6, 7], the form deviation from tip radius was specified as 4 µm for the working grade [6], and 2 µm for the calibration grade [7] Rockwell indenters. The large tolerances specified for the form deviation could result in either flat- or sharp-shaped Rockwell indenters. This could generate large variations in Rockwell hardness tests, especially for lower force Rockwell hardness tests using diamond indenters (i.e., HR45N, HR30N and HR15N scales).

3. INVESTIGATION OF THE INFLUENCE OF FORM DEVIATIONS ON ROCKWELL HARDNESS TESTS

The spherical tip of a Rockwell diamond indenter tends to be manufactured as either a flat- or sharp-shaped surface because of the anisotropic property of the diamond. Fig. 2a shows a flat-shaped indenter. After least squares arc fitting, the form deviation shows a central valley of 0.76 µm (see Fig. 2b). Fig. 2c shows a sharp-shaped indenter. After least squares arc fitting, the form deviation shows a central peak of 0.82 µm (see Fig. 2d). Even both indenters having a close tip radius and having a form deviation less than ± 1 µm, their flat and sharp shape can be obviously seen from Fig. 2a and 2c. If their profile deviations were increased to ± 2 µm, there must be a large difference in both their geometry shapes and hardness performances.

The purpose of specifying tolerances for form deviations from the tip radius is to ensure the correct shape and the performance uniformity of Rockwell indenters, which was specified as ± 0.4 HR for the calibration grade Rockwell indenters (see Table 1) [7]. In order to investigate the effect of form deviation on HRC tests, two sets of previous measurement data were analyzed. First, a set of nine Rockwell indenters was measured at NIST using a stylus instrument. The mean tip radii ranged from 198.34 µm to 202.90 µm, and the mean cone angles ranged from 119.94° to 120.07° [2]. The average profile deviations ranged from 0.47 µm (maximum peak) to 0.41 µm (maximum valley). The NIST expanded measurement uncertainty is shown in Table 1. These nine indenters were combined with two other indenters previously measured at NIST with close geometries. These eleven indenters were tested using the NIST standard deadweight machine. The test results showed a total performance variation range of – 0.17 HRC to + 0.23 HRC over a range of 25 HRC to 60 HRC for all 11 indenters [2].

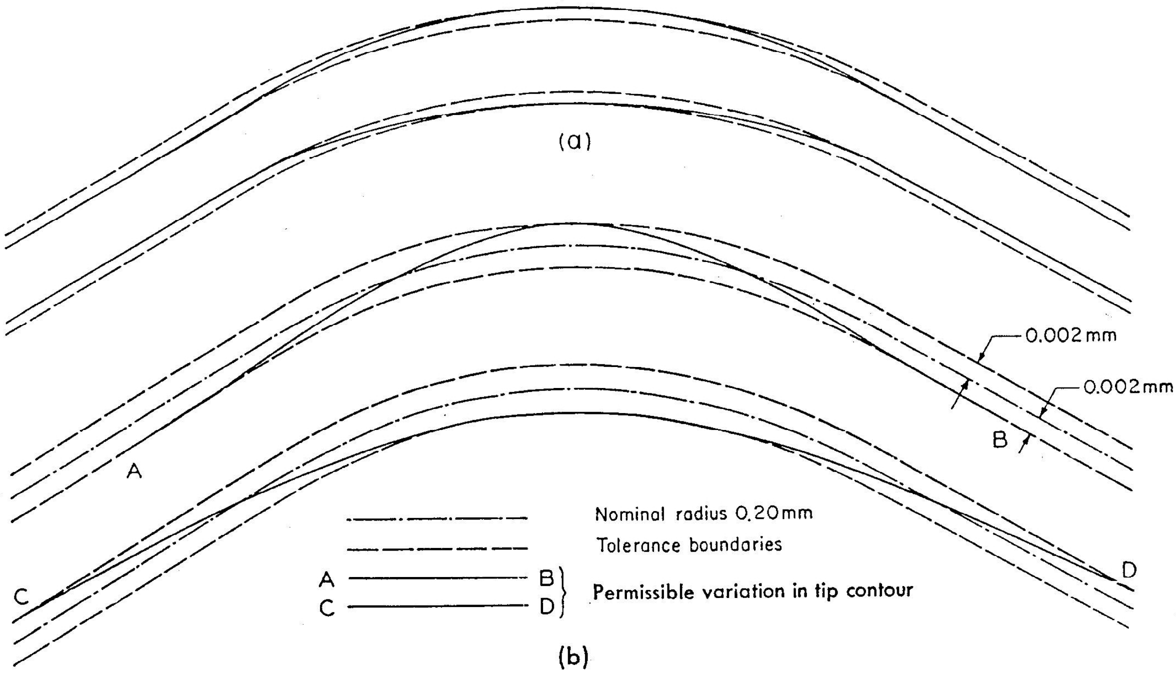

Fig. 1. Tolerance on contour of diamond sphero-conical penetrator [8, 9], (a) 500×; (b) not to scale.

As a comparison, a second set of data was analyzed from tests using four NIST indenters and five NMIJ (National Metrology Institute of Japan) indenters, all measured at NIST using the same stylus instrument. Their microform geometry parameters were very close to those of the first set of 11 indenters. The mean tip radii of the second set of nine indenters ranged from 197.25 µm to 202.43 µm, and the mean cone angles ranged from 119.89° to 120.21° [2]. As to the form deviations of the second set of nine indenters, eight of them were very close to the first set of indenters. Their average form deviations ranged from 0.33 µm (maximum peak) to 0.38 µm (maximum valley). However, one indenter in the second set showed significant bias in form deviation with respect to the other eight indenters. Its maximum peak deviation was 0.92 µm, which was significantly larger than the other eight indenters [2].

For all nine indenters of the second set, the hardness performance tests showed a total variation range of – 0.23 HRC to + 0.32 HRC over a range of 25 HRC to 63 HRC. This variation range was larger than that of the first set of 11indenters, from – 0.17 HRC to + 0.23 HRC. However, one indenter in the second set with large form deviation (0.92 µm) showed performance bias with respect to the other eight indenters [2]. When the data for this indenter is removed, the other eight indenters showed a performance variation range of ± 0.19 HRC with respect to the recalculated means, which is close to the first set of 11 indenters.

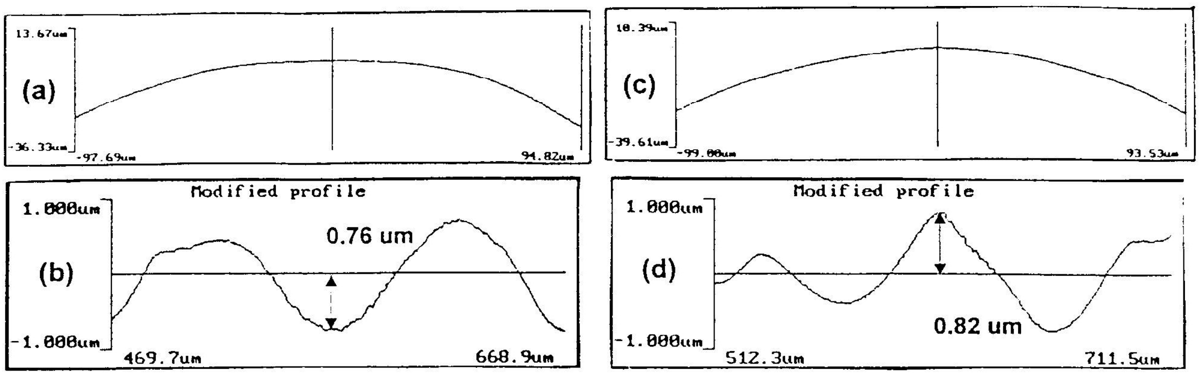

Fig. 2. Flat- and sharp-shaped Rockwell diamond indenters: a) A flat-shaped indenter; b) After least squares arc fitting, a 0.76 µm central profile valley can be seen; c) A sharp-shaped indenter; d) After least squares arc fitting, a 0.82 µm central profile peak can be seen.

From the analysis of two sets of data shown above, it suggests that a 0.92 µm form deviation may be too large for a calibration grade Rockwell indenter. It may generate a significant performance variation with respect to the other indenters having a ± 0.5 µm form deviation range. And a ± 0.5 µm form deviation may be suitable for the calibration grade Rockwell indenters, as well as for the NIST SRM indenters.

The Finite Element Analysis (FEA) simulation results showed that, for HRC tests of soft materials at 25 HRC level, form deviation of the Rockwell indenter had a very small effect [13]. That was in accordance with previous experimental results and conclusions [14], because the entire tip of the Rockwell indenter was in full contact with the soft material even during the preliminary testing force period (98N). However, form deviations showed significant effect on HRC tests in 45 HRC and 64 HRC levels. From Fig. 3, it can be seen that when the Rockwell indenter is sharp-shaped, and if the central profile peak deviation increased to 2 µm (see Fig. 3, right), that could cause about 0.8 HRC increase in hardness test at 64 HRC level, and about 0.6 HRC increase in HRC tests at 45 HRC level. FEA simulation results also showed that when the Rockwell indenter is flat-shaped, and if the central profile valley increased in depth (see Fig 3, left), that could cause HRC readings to decrease. But the effect is not as large as that of the profile peak. This is also in accordance with previous experimental results and conclusions that Rockwell indenters with sharp tips affect HRC readings more significantly than those with flat tips [14].

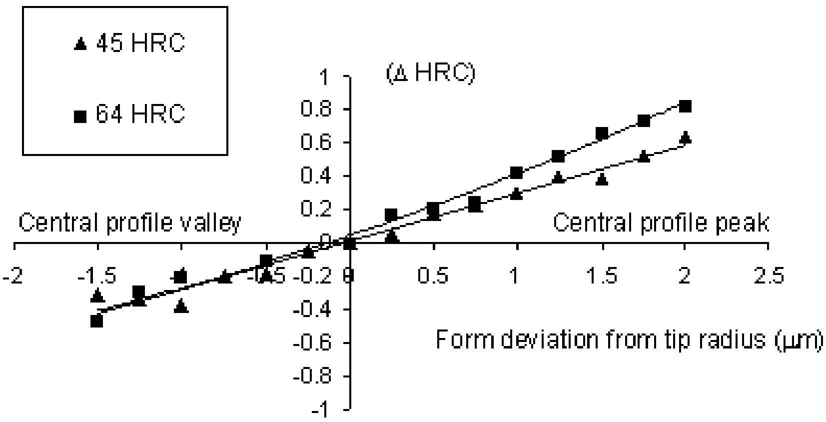

Fig. 3. HRC variations at HRC 45 and HRC 64 level with change of form errors

4. PROPOSED TOLERANCE FOR FORM DEVIATIONS OF NIST SRM 2809 ROCKWELL DIAMOND INDENTERS

For the calibration grade Rockwell diamond indenters, it is required that the hardness performance uniformity with respect to a Standard Indenter is within ± 0.4 HRC (see Table 1). From Fig. 3, it can be seen that ± 0.5 µm form deviation may generate about ± 0.2 HRC variation at 64 HRC level. And either ± 1 µm or ± 2 µm form deviation may be too large to meet the requirement of hardness performance uniformity for the calibration grade Rockwell diamond indenters. Based on the analyses of previous test data and FEA simulation results, we specified ± 0.5 µm form deviation tolerance for the production of NIST SRM 2809 Rockwell Diamond Indenters.

The surface finish roughness of Rockwell diamond indenters also affects hardness performance [8], and therefore, needs to be controlled. It is proposed to measure the roughness average *Ra* [17] for control of the surface roughness of NIST SRM Rockwell indenters. The tolerance is specified as 0.005 µm for the mean *Ra* value and 0.006 µm for the maximum *Ra* value.

5. STANDARD ROCKWELL DIAMOND INDENTERS

Besides the working and calibration grade Rockwell indenters specified in ASTM and ISO standards [5-7], NIST also proposed a "Standard Grade" Rockwell indenter [2], which is used at National Metrology Institutes (NMI's) as a Primary National Standard for maintaining the National Rockwell Hardness Scale, and for international comparison to establish a worldwide unified Rockwell hardness scale [2]. The Standard Rockwell Indenters are characterized by very high uniformity in both their microform geometries and hardness performances [2]. The geometrical tolerances are proposed as 50 % of the tolerances of the calibration grade indenters specified in ASTM and ISO standards [5, 7], except the form deviation from the tip radius, which is proposed as ± 0.25 µm [2]. The hardness performance uniformity is proposed as ± 0.15 HRC within a group of geometrically qualified Standard Indenters [2]. The indirect verification of both the calibration grade and the working grade Rockwell indenters are performed by hardness comparisons with respect to the Standard Indenters [5-7].

6. ADVANCES IN DEVELOPING NIST STANDARD AND SRM ROCKWELL DIAMOND INDENTERS

At NIST, a Microform Calibration System was established in 1994 with sufficiently high measurement accuracy to support the microform calibrations of the Standard Rockwell Diamond Indenters [15]. The combined measurement uncertainty was ± 0.3 µm for the 200 µm tip radii measurements, and ± 0.01E for the 120E cone angle measurements, both with a coverage factor of k = 2. In April, 1995, a group of 11 Rockwell indenters was calibrated by this system. These indenters showed a variation range of the mean least squares radii from 198.34 µm to 202.90 µm, and a variation range of the mean cone angles from 119.94E to 120.07E [2]. These geometrical parameters are very close to the technical requirements of the NIST proposed Standard Rockwell Indenters: 200 µm ± 2.5 µm and 120E± 0.05E. However, quality problems of these indenters come from the profile peak and valley deviations (about ± 0.5 µm), as well as from the cone flank straightness (about 0.5 µm). For the proposed Standard Rockwell Indenters, both technical requirements are specified within a tolerance range of ± 0.25 µm and 0.25 µm [2].

The 11 indenters were performance-tested at the NIST deadweight standardized hardness machine over nominal hardness values ranging from 25 HRC to 60 HRC. The deviations from the measured averages ranged from – 0.17 HRC to + 0.23 HRC. This variability in measured performance also included the random variations of the standard hardness machine and the random non-uniformity of the standard hardness blocks. These results suggest that a qualified set of Standard Indenters could produce a hardness measurement uniformity of about ± 0.15 HRC, by which the worldwide HRC scales could probably be unified to ± 0.2 HRC without significant bias to an ideal scale [2]. Furthermore, international comparisons showed that the standard indenters could be produced by different diamond manufacturers and calibrated by different measurement techniques with sufficient uniformity both in their microform geometries and hardness performances [2].

In 1995, one of the 11 NIST Rockwell indenters, numbered 3581, was selected as the NIST Primary Rockwell Indenter for establishing and maintaining the U.S. National Rockwell C Hardness Scale. After more than 3000 hardness indentations with the maximum loading of 1471 N for the calibration of SRM 2810 - 2812 Rockwell C hardness blocks, this indenter was recalibrated in 1997. In 2004, the Microform Calibration System was moved to the Advanced Measurement Laboratory (AML) at NIST; some upgrades on the hardware and software of the measurement system were completed in 2005. The No. 3581 indenter was recalibrated in 2005. The calibration results showed high reproducibility with the previous results. For example, the nominal 200 µm mean least squares radius was calibrated as (199.06 ± 1.97) µm in 1995, (199.24 ± 1.19) µm in 1997, and (199.11 ± 1.30) µm in 2005. The nominal 120˚ cone angle was calibrated as (119.995 ± 0.020)E in 1995, (120.012 ± 0.017)E in 1997, and (119.987 ± 0.020)E in 2005. The profile deviation and cone straightness parameters also showed very high reproducibility [16]. These calibration results demonstrated the high stability and long-term reproducibility for both the NIST Primary Rockwell Indenter and the NIST Microform Calibration System for support of the U.S. National Rockwell C Hardness Scale, and for the establishment of a worldwide unified Rockwell hardness scale with metrological traceability.

7. SUMMARY

NIST is developing Standard Reference Material (SRM) 2809 Rockwell Diamond Indenter to support Rockwell hardness standardization. The SRM indenters are characterized by geometric and hardness performance uniformity, measurement traceability, stability and interchangebility. Most tolerances for the SRM indenters are adopted from those of the calibration grade indenters specified in ASTM and ISO standards [5-7]. However, based on the data of geometrical measurements and hardness tests at NIST, and the results of FEA simulations, it is proposed to use ± 0.5 µm tolerance as the form deviation from the tip radius for the production of NIST SRM Rockwell diamond indenters.

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