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DEVELOPMENT OF NIST STANDARD REFERENCE METERIAL (SRM) ROCKWELL HARDNESS DIAMOND INDENTERS

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Abstract: The National Institute of Standards and Technology (NIST) is developing Standard Reference Material (SRM) Rockwell Hardness Diamond Indenters to support Rockwell hardness standardization in the U.S. One of the key steps is the geometrical calibration of the SRM indenters. Most tolerances for the geometrical parameters of the SRM indenters are adopted from those of the calibration grade Rockwell diamond indenters specified in the ASTM and ISO standards except for the form deviation from tip radius which was specified with a smaller tolerance of ± 0.8 µm. From the initial calibration results of the SRM indenters, it was found that the 0.5 µm tolerance for the cone flank straightness specified for the calibration grade diamond indenters by the ISO standard might be too tight to fit in the current production capability of the diamond manufacturers in different countries. As a result, although most SRM diamond indenters calibrated at NIST could meet all the technical requirements including the ± 0.8 µm reduced tolerance for the form deviation from the tip radius, none could meet the 0.5 µm tolerance of the cone flank straightness specified in the ISO standard when the calibrations were performed under the nominal window size. Furthermore, the window size and location for the geometrical calibration of the Rockwell diamond indenters are not clearly specified in the current standards, that also makes the geometrical calibration of Rockwell diamond indenter more difficult.

Keywords: Rockwell hardness, diamond indenter, form deviation, microform calibration, standard reference material.

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 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" is used, the hardness variation range is much less than that of using different national indenters [3].

The National Institute of Standards and Technology (NIST) is developing Standard Reference Material (SRM) Rockwell Hardness Diamond Indenter to support Rockwell hardness standardization in the U.S. One of the key steps is the geometrical calibration of the SRM indenters. In this paper, we describe the geometrical specification of the SRM indenters and the capability of the NIST microform calibration system in Section 2 and 3, and discuss the initial calibration results and suggestions in Section 4.

2. GEOMETRICAL SPECIFICTIONS FOR NIST SRM ROCKWLL DIAMOND INDENTERS

The Rockwell hardness diamond indenter is made of 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.

Two grades of Rockwell diamond indenters, working grade and calibration grade, are specified in the ASTM [4,5] and ISO standards [6,7]. 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 HRC hardness blocks. For the 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 the precise tip radius [8], a tighter tolerance, 0.8 µm was specified for the form deviations from the mean radius [10], instead of the 2 µm tolerance specified in ISO and ASTM standards. Furthermore, because the surface roughness of diamond indenters affects hardness tests [5, 6], a tolerance for the surface finish roughness Ra was specified for the SRM indenters [10].

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 C hardness (HRC) tests [2]. The development of Standard Reference Material (SRM) Rockwell diamond indenter with accurately calibrated geometrical parameters is an important step towards Rockwell hardness standardization in the U.S. In 1994, NIST developed a microform calibration system based on a stylus instrument 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 [3,10]. 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 [10], 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 geometrical calibration for 28 NIST SRM diamond indenters showed that, the cone flank straightness was a key quality issue. It was found that the 0.5 µm tolerance of cone flank straightness specified in the current ISO standard [7] might be too tight to fit in the current production capability of the diamond manufacturers in different countries. As a result, although most SRM diamond indenters calibrated at NIST could meet all the technical requirements including the ± 0.8 µm reduced tolerance for the form deviation from the tip radius, none could meet the 0.5 µm tolerance of the cone flank straightness specified in the ISO standard when the calibrations were performed under the nominal window size. This is a consequence of the technical specification of the current ISO standards [7], in which a tight tolerance, 0.5 µm, is specified for the cone flank straightness.

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 International Committee for Weights and Measures (CIPM) Key Comparisons (KC) for international Rockwell hardness tests of the Working Group on Hardness (WGH) in the framework of the Consultative Committee for Mass and Related Quantities (CCM).

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