Metrological Characterization of Hardness Indenter Calibration System

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Abstract. There are four main components constituting the conventional hardness scales, in all types of hardness measurements, from testing machines to standardizing ones: Force application, indentation size measurement, indenter and measurement cycle. To assure the quality, reliability and uniformity in hardness measurement in all over the world it is important to constitute traceability of each parameter to the base SI units and confirm the performance of the system as a whole. One of the most interesting subject to be discussed is the calibration of the geometrical properties of the diamond indenters used in Rockwell and Vickers scales, spherico-conical Rockwell and square-based pyramid Vickers diamond indenters. The indenters to be used in testing and calibration/standardizing machines are supposed to be in accordance with some international hardness standards, namely ISO 6507-2 [5] and ISO 6508-2 [2] for testing Vickers and Rockwell machines, respectively; ISO 6507-3 [6] and ISO 6508-3 [3] for Vickers and Rockwell calibration/standardizing machines, respectively, and relevant ASTM standards. This paper describes the metrological characterization of UME Hardness Diamond Indenters Calibration System and the traceability of each measured parameter subject to calibration.

1. Introduction

Hardness is a widely used material property and defined as the resistance to deformation. Regarding its definition it is handled as comprising two main steps: Realization of deformation and measurement of the size of deformation. To standardize hardness measurements in all over the world it is important to standardize all components constituting each hardness scale, such as force applied, testing cycle through which the indentation is realized, indentation measurement and indenters used to realize the deformation.

One of the main issues in the world of Hardness Metrology is the standardization of indenters and it has been discussed for long time how to make well defined calibration of dimensional parameters of the Rockwell and Vickers diamond indenters. Due to their small size, difficulties because of their geometry and material properties such as crystallographic structure of diamond, their calibration becomes relatively difficult and time consuming, compared to ordinary dimensional calibrations. The indenter geometry and the tolerances within which they are supposed to be are defined in the relevant hardness standards such as ISO 6507-2/3 [5][6], ISO 6508-2/3 [2][3] and relevant ASTM [7][8] standards. The geometrical properties of the indenters are critical and have significant effects on their penetration performance and in turn measurement results.

The system used for calibration of Rockwell and Vickers diamond indenters in UME Hardness Laboratory is composed of two parts. The first part is composed of a metallographic microscope with some objective lenses with double-beam (Mirau) interferometry and angular encoder assembly. In this part of the system angle and tilt angle (Rockwell-Vickers); straightness (Rockwell); squareness, flatness and length of line of junction (Vickers) is measured. The second part consists of a rotating table centered on air bearings. Here the Rockwell indenters are mounted on the table and along the
rotation of the table, the indenter tip profile is detected by a linear transducer and radius and deviation between the actual profile and the best-fitting spherical profile (shape error) is measured.

2. Characterization of the System

To have a better understanding of the system performance, we made use of the some artifacts to be used as transfer standards and mounted on to the system as to have the same function as the indenters for every calibration parameter. We made the calibration of the system and tried to decrease its uncertainty in the way explained below and constituted its traceability. In evaluation of the measurement results we calculated the uncertainty of the system and its deviation (error) from the reference values and we determined the tolerances (Tol) within which the system is available for use.

The tolerance of the Hardness Indenter Calibration System (HICS) is the summation of the measurement uncertainty and the error (absolute) and calculated by

\[ Tol = \text{Abs}(E) + U_{MEAS} \] (1)

2.1. Radius and shape error measurements

The Rockwell diamond indenters are supposed to have a cone with a nominal radius of 0.200 mm at the tip with an average of ±5 μm tolerance in accordance with ISO 6508-3:2015 [3]. To check the performance of the system in radius measurements we adopted 3 ruby spheres with radius values of 203.02 μm, 201.87 μm and 194.51 μm such that covers the range of the tolerances requested by ISO 6508-3:2015 [3] as much as possible. Also we have the spheres calibrated for an arc of ±30° angle of radius with an uncertainty of 0.350 μm for radius measurements (figure 1-a/b/c) and an uncertainty of 0.050 μm for shape error measurements (figure 1-d) for 8 sections. These calibrations were carried out on an ultra-precise coordinate measuring machine (μ-CMM) used for calibrating ultra small objects in 3D. Both, calibration of the spheres and characterization of the Hardness Indenter Calibration System (HICS) is performed in the same way; for the same section (figure 1-a) and same arc-radius (figure 1-b).

![Figure 1. Calibration of the Reference Ruby Spheres and Calibration of HICS](image)

The HICS is adjusted with a reference ruby that is one section of the 202.55 μm radius (at 0° profile) of the first sphere. Then all sections of the 3 ruby spheres are measured. The result for one sphere of every section is given below in Table 1.

| Profile | UME / μm | Ref / μm | Error (E) / μm | U_{ref} / μm | U_{MEAS} / μm | Tol / μm |
|---------|----------|----------|----------------|-------------|--------------|----------|
| 0°      | 0.02     | 0.051    | -0.04          | 0.03        | 0.05         | 0.39     | 0.08 |
| 22.5°   | 0.10     | 0.060    | -0.20          | 0.04        | 0.05         | 0.35     | 0.05 |
| 45°     | 0.09     | 0.069    | -0.24          | 0.02        | 0.05         | 0.35     | 0.05 |
| 67.5°   | 0.11     | 0.093    | -0.18          | 0.01        | 0.05         | 0.35     | 0.05 |
| 90°     | 0.07     | 0.062    | 0.17           | 0.03        | 0.05         | 0.35     | 0.05 |
| 112,5°  | 0.10     | 0.054    | 0.23           | 0.05        | 0.05         | 0.35     | 0.05 |
| 135°    | 0.09     | 0.053    | 0.16           | 0.03        | 0.05         | 0.35     | 0.05 |
| 157.5°  | 0.10     | 0.071    | 0.10           | 0.03        | 0.05         | 0.35     | 0.05 |

Table 1. Radius (R) and Shape Error (SE) Measurements for 8 Sections of One Ruby Sphere
We calculated the average values of each sphere by making use of the 8 sections the results are given in Table 2.

Table 2. Radius (R) and Shape Error (SE) Measurements for Average Values Three Ruby Spheres

| Spheres | UME / μm | Ref / μm | Error / μm | U_rel / μm | U_MEAS / μm | Tol / μm |
|---------|----------|----------|------------|------------|-------------|----------|
| Sphere-1 | 203.00   | 0.09     | 0.06       | 0.02       | 0.03        | 0.465    |
| Sphere-2 | 202.30   | 0.11     | 0.06       | 0.30       | 0.05        | 0.373    |
| Sphere-3 | 194.92   | 0.13     | 0.05       | 0.41       | 0.07        | 0.351    |

2.2. Angle and tilt angle measurements

The angle measurement of The Hardness Indenter Angle Calibration Gauge (see figure 2) was performed by using ZEISS Prismo 7 S-ACC CMM along the measurement surface. The measurement surfaces of the gauges are divided into 3 regions as the upper region, the middle region and the lower region, and the angle of the calibrating surface and the upper and lower regions are defined as 3 mm up and 3 mm below according to mid point. The result of each region is calculated with the results of the corresponding region. (Side 1 lower region (red line) and Side 2 lower region (red line), Side 1 middle region (blue line) and Side 2 middle region (blue line), Side 1 upper region (yellow line) and Side 2 upper region (yellow line) as shown in Figure 2 and the measurement results are given in Table 3.

Table 3. Angle Measurements for Rockwell and Vickers Diamond Indenters

| Angle G. | UME / °  | Ref / °  | Error / °  | U_rel / °  | U_MEAS / ° | Tol / °  |
|----------|----------|----------|------------|------------|------------|----------|
| Gauge-1  | 118.9500 | 118.9421 | 0.01       | 0.003      | 0.007      | 0.01     |
| Gauge-2  | 119.5349 | 119.5393 | 0.00       | 0.003      | 0.007      | 0.01     |
| Gauge-3  | 119.6482 | 119.6364 | 0.01       | 0.003      | 0.007      | 0.02     |
| Gauge-4  | 119.9982 | 119.9909 | 0.01       | 0.003      | 0.007      | 0.01     |
| Gauge-5  | 120.0358 | 120.0203 | 0.02       | 0.003      | 0.007      | 0.02     |
| Gauge-6  | 135.9958 | 136.0136 | -0.02      | 0.003      | 0.007      | 0.02     |
| Gauge-7  | 135.9804 | 135.9738 | 0.01       | 0.003      | 0.007      | 0.01     |
| Gauge-8  | 135.7424 | 135.7527 | -0.01      | 0.003      | 0.007      | 0.02     |
| Gauge-9  | 135.4477 | 135.4430 | 0.00       | 0.003      | 0.007      | 0.01     |
| Gauge-10 | 135.6781 | 135.6627 | 0.02       | 0.003      | 0.007      | 0.02     |

2.3. Squareness measurements

The squareness measurement of Angle Calibration Gauge from the four sides were performed again by using ZEISS Prismo 7 S-ACC CMM along the midpoint of measurement surfaces. Angle calculations between the surfaces were made according to each other. Then the same gauge is used for the calibration of HICS and the measurement results are given in Table 4.

Table 4. Squareness Measurements for Vickers Diamond Indenters

| Section | UME / °  | Ref / °  | U_rel / °  | Error / °  | U_MEAS / ° | Tol / °  |
|---------|----------|----------|------------|------------|------------|----------|
| 1 - 2   | 87,8241  | 87,8301  | 0.0028     | -0.0060    | 0.0057     | 0.012    |
| 2 - 3   | 89,8816  | 89,8916  | -0.0100    | 0.0059     | 0.016      |
| 3 - 4   | 87,6343  | 87,6180  | 0.0163     | 0.0037     | 0.020      |
| 4 - 1   | 94,6604  | 94,6708  | -0.0104    | 0.0034     | 0.014      |
2.4. Flatness & straightness measurements

In the system flatness for the Vickers indenters 4 faces is measured. In this measurement the objective lenses with double-beam (Mirau) interferometry is used. A high quality gauge block for a good surface uniformity is used for flatness measurement. The gauge block is calibrated in UME Dimensional Laboratory. These block’s flatness is measured by the Hardness Indenter Calibration System. The indenters’ straightness value shall not exceed 0.7 \( \mu \text{m} \) for Rockwell indenters and flatness shall not exceed 0.5 \( \mu \text{m} \) for Vickers indenters. The following results are found over 1 mm length.

Table 5. Flatness Measurements for Vickers Diamond Indenters

| Meas. / \( \mu \text{m} \) | UME / \( \mu \text{m} \) | Ref / \( \mu \text{m} \) | Error / \( \mu \text{m} \) | U_{MEAS} / \( \mu \text{m} \) | Tol / \( \mu \text{m} \) |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 0.057           | 0.065           | 0.029           | 0.036           | 0.021           | 0.057           |
| 0.069           | 0.069           | 0.029           | 0.036           | 0.021           | 0.057           |
| 0.063           | 0.063           | 0.029           | 0.036           | 0.021           | 0.057           |

2.5. Length of line of junction measurements

The Vickers indenters 4 surfaces meet at one point at the tip but they sometimes make a roof line which is requested to be as small as possible, the best is zero. It needs to be measured and in this system it is measured by 100X lens with some crosswires calibrated by a length reference like stage micrometer.

The smallest length increment we have is 10 \( \mu \text{m} \) and we calibrated the system for (0 – 50) \( \mu \text{m} \) range and the found result is given below.

Table 6. 100X Objective Lens Legth Measurement Result

| Nom. / \( \mu \text{m} \) | Ref / \( \mu \text{m} \) | UME / \( \mu \text{m} \) | Error / \( \mu \text{m} \) | U_{MEAS} / \( \mu \text{m} \) | Tol / \( \mu \text{m} \) |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| 0               | 0.00            | 0.00            | 0.00            | 0.00            | 0.00            |
| 10              | 10.05           | 10.13           | 0.08            | 0.17            | 0.25            |
| 20              | 20.06           | 20.17           | 0.11            | 0.17            | 0.27            |
| 30              | 30.06           | 30.17           | 0.11            | 0.17            | 0.27            |
| 40              | 40.04           | 40.20           | 0.16            | 0.15            | 0.31            |
| 50              | 50.02           | 50.20           | 0.18            | 0.15            | 0.33            |

3. Conclusion

At the end of this study the traceability in measurement of the main parameters such as angle and tilt angle (Rockwell-Vickers); flatness, squareness and length of line of junction (Vickers); radius, straightness and deviation between the actual profile and the best-fitting spherical profile (shape error) (Rockwell) is constituted. The measurement uncertainty due to the Hardness Indenter Calibration System is decreased down to 0.02\(^\circ\) for angle, tilt angle and squareness measurements, to 0.60 \( \mu \text{m} \) for radius measurements, to 0.13 \( \mu \text{m} \) for shape error measurements and 0.06 \( \mu \text{m} \) flatness measurements.

4. References

[1] ISO 6508-1: Metallic Materials – Rockwell Hardness Test – Part1: Test Method (scales A, B, C, D, E, F, G, H, K, N, T), ISO, 2016.
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[3] ISO 6508-3: Metallic Materials – Rockwell Hardness Test – Part3: Calibration of Reference Blocks (scales A, B, C, D, E, F, G, H, K, N, T), ISO, 2015.
[4] ISO 6507-1: Metallic Materials – Vickers Hardness Test – Part1: Test Method, ISO, 2005.
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[7] ASTM E18:Standard Test Methods for Rockwell Hardness of Metallic Materials, ASTM, 2017.
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