

## DETERMINATION OF THE GEOMETRICAL PARAMETERS OF ROCKWELL DIAMOND INDENTERS BY ITERATIVE REGRESSION METHOD

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**Abstract:** For the verification of Rockwell diamond indenter exactly following the international definition, an iterative method with the least square circle fitting was introduced. This method was applied to the analysis of verification data obtained with a laser probe 3D profile measurement instrument. It was demonstrated that the technique can be used to express the geometrical parameters properly with analytical results as an example. In addition, this technique can be applicable to determine equivalent geometrical parameters obtained with the optical measurement system currently used at NMIJ to establish the national standard indenters. It is shown that the proposed method can describe the geometry of indenter better than currently used method. These results suggest that the uncertainty of the national standard indenter could be improved through this high resolution geometry measurement and the multiple regression analysis NMIJ has been using.

**Keywords:** Rockwell diamond indenter, tip radius, cone angle, laser probe 3D profile measurement instrument, least square circle fitting.

### 1. INTRODUCTION

It is generally recognized that the geometrical error of diamond indenter affects significantly to indicated hardness values in Rockwell hardness measurement and therefore many efforts regarding indenter verification has been made for many years. Various types of instruments have been used for the geometrical verification by many researchers, *e. g.*, stylus-type surface profiler[1], micro-range coordinate-measuring machine ( $\mu$ CMM)[2], specially designed for spherical and conical parts of Rockwell diamond indenter with combination of interferometric optics and precise positioning mechanics [3, 4, 5], *etc.* Recently, observation techniques in small objects have been remarkably improved and 3D geometry of small objects has been able to obtain easier and more accurate. 3D optical surface profiler with the white light interferometer [6], confocal laser scanning microscope [7] and very wide range atomic force microscope [8] were used to observe precise 3D image of Rockwell diamond indenters. Along with those investigations, the effects of geometrical error to hardness values are also studied by multiple regression analysis [3, 9] or the method using finite element analysis [8,10]. With

those backgrounds, a pilot study to investigate international metrological equivalency of Rockwell diamond indenters is now in progress under the framework of the International Committee for Weights and Measures (CIPM)[11].

The verification of the indenter geometry has a meaning to confirm how accurately the indenter geometry follows its defined shape. It is commonly believed that smaller geometrical error brings more certain value of hardness. It is, however, still difficult to improve the quality of indenter because the manufacturing technique is not much changing as the measuring techniques. From this reason, the correction of hardness value for each indenter have been considered [12]. Yano *et al.* proposed unique technique to estimate the bias of each indenter from the hardness value which would be realized by the ideally shaped indenter by means of the multiple regression analysis between geometric parameters of indenter and hardness [3] and it have been formal procedure to establish the national Rockwell hardness standard in National Metrology Institute of Japan (NMIJ) until now. In order to utilize this technique, it is important to evaluate appropriate geometric parameters from the verification results of indenters.

In this paper, the analytical method to obtain desirable geometric parameters is introduced from the measurement data of a Rockwell diamond indenter obtained with a laser probe 3D profile measurement instrument. It allows us to verify an indenter exactly compliant to the definition of CIPM and also compatible with currently used method at NMIJ. The efficiency of proposed analytical method was demonstrated with obtained verification results.

### 2. MEASUREMENT OF INDENTER GEOMETRY

The geometry of each Rockwell diamond indenter is measured with a laser probe 3D profile measurement instrument shown in Fig. 1 (Model NH3-SP, Mitaka Koki, Co. Ltd., Japan)[13]. This instrument can detect *z*-axis coordinate with an auto-focus mechanism with probe laser beam though the lens during scanning of sample on the movable sample stage. The resolutions are 0.01  $\mu$ m in *z*-axis with 100 $\times$  objective lens and 0.1  $\mu$ m in *x*- and *y*-axes. One of the advantages of this instrument is that measurable range is not limited in the field of view of the microscope. In addition, it has a capability to set an arbitrary coordinate system for measurement independently from its mechanical layout. It enables us to set the datum plane for indenter

measurement. In this study, the datum plane was set on the seating face of the indenter. Due to this function, it is also possible to evaluate the tilt of indenter axis. The setup of a Rockwell diamond indenter on the  $xy$  stage of the instrument is shown in Fig. 2. Profiles of a Rockwell diamond indenter are measured in four cross sections parallel to the axis in every  $45^\circ$ . The result is graphically shown in Fig. 3. In this



Fig. 1 The laser probe 3D profile measurement instrument used to measure the cross section of Rockwell diamond indenters (courtesy of Mitaka Koki, Co. Ltd.).

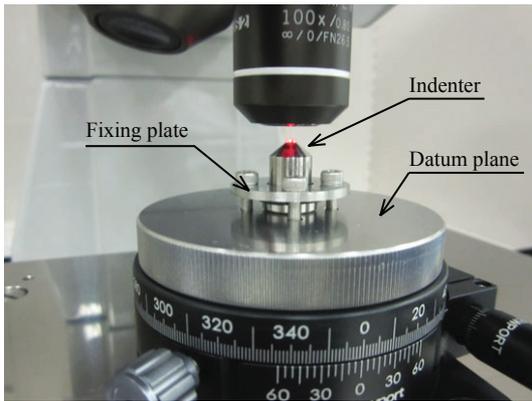


Fig. 2 Setup of a Rockwell diamond indenter for the datum plane based profile measurement.

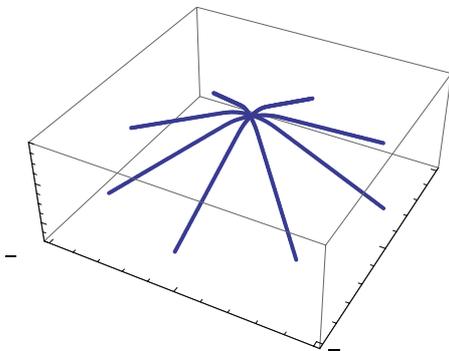


Fig. 3 Measurement result of a Rockwell diamond indenter in the horizontal range of  $1\text{ mm} \times 1\text{ mm}$ .

Table 1 The definition of the geometry of Rockwell diamond indenter by CIPM/CCM/WGH.

Parameters	Reference value	Start measurement	Stop measurement
Cone angle, $\alpha_m$	$120^\circ$	$\pm 30^\circ$	$\pm 400\ \mu\text{m}$
Tip radius, $R_a$	$200\ \mu\text{m}$	$-30^\circ$ <sup>†</sup>	$+30^\circ$ <sup>†</sup>

<sup>†</sup> from the axis

case, the tilt of the datum plane was  $0.03^\circ$  and this was automatically corrected by the instrument.

### 3. ANALYTICAL METHOD OF THE GEOMETRY

The geometry of Rockwell diamond indenter was defined by the Working Group on hardness (WGH), Consultative Committee for Mass and Related Quantities (CCM), CIPM as Table 1[14].

In order to verify an indenter according to the definition, it is important to determine the blend points between cone and sphere from measured profile properly. The author developed an analytical method to determine the coordinate of these points with an iterative calculation. Its flowchart is illustrated in Fig. 4.

#### 3.1 Extraction of the dataset to be analysed in the spherical part of indenter

For the verification of the spherical part of an indenter, the dataset of corresponding part should be extracted from the whole profile data in the central angle range between  $-\phi_t/2$  ( $\mathbf{P}_1^{(m)}$ ) and  $\phi_t/2$  ( $\mathbf{P}_2^{(m)}$ ), whereas  $\phi_t = 60^\circ$  in WGH/ISO compliant verification (see Fig. 5). The symbol  $m$  in the parentheses represents the number of the iteration. In the beginning of the iterative calculation, tentative center of curvature ( $\mathbf{P}_0^{(0)}$ ) is needed to determine.

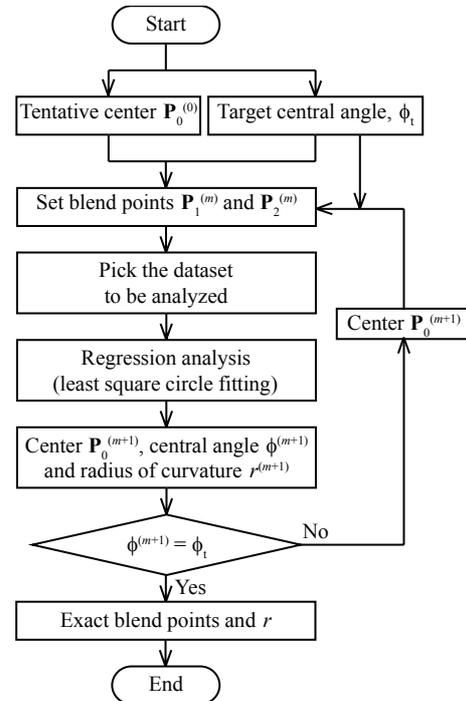


Fig. 4 Flowchart for the determination of the tip radius  $R_a$  and the positions of blend points.

### 3.2 Least square circle fitting

There are several methods to fit a circles to data[15]. Among them, the simplest and the most primitive way is chosen for this analysis. In order to fit the data to the equation

$$(x-a^{(m)})^2 + (z-b^{(m)})^2 = r^{(m)2}, \quad (1)$$

the following function can be taken as the measure of the fitting,

$$SS(a^{(m)}, b^{(m)}, r^{(m)}) = \sum_{i=1}^N \left( r^{(m)} - \sqrt{(x_i - a^{(m)})^2 + (z_i - b^{(m)})^2} \right)^2 \quad (2)$$

Following the principle of the least square, the constants,  $a^{(m)}$ ,  $b^{(m)}$  and  $r^{(m)}$  are determined when Equation (2) is minimized.

### 3.3 Calculation of the central angle of the spherical part

After the least square circle fitting analysis, different point of the center of the spherical part is obtained from the previous iteration or the initially estimated position. It brings different central angle expressed by the following equation,

$$\phi^{(m)} = \tan^{-1} \frac{z_N^{(m)} - b^{(m)}}{x_N^{(m)} - a^{(m)}} - \tan^{-1} \frac{z_1^{(m)} - b^{(m)}}{x_1^{(m)} - a^{(m)}}. \quad (3)$$

where  $N$  is the number of data points in selected dataset.

### 3.4 Iteration

If  $\phi^{(m)} \neq \phi_t$ , pick different dataset according to the procedure described in 3.1 with parameters  $a^{(m)}$  and  $b^{(m)}$  and carry out the analysis. When  $\phi^{(m+1)}$  agrees with  $\phi_t$  within the resolution of the dataset, the best evaluation of the spherical part of an indenter is obtained.

### 3.5 Convergence criterion

An iterative calculation does not always bring the right answer but it works when the convergence criterion is satisfied. Let the deviation of the calculated central angle  $\phi^{(m)}$  from the target angle  $\phi_t$  as

$$F(\phi^{(m)}) \equiv \phi^{(m)} - \phi_t. \quad (4)$$

When the solution is found, Equation (4) goes zero.

As seen in Equation (3),  $\phi^{(m)}$  is the function with 6 parameters, *i. e.*,

$$\phi^{(m)} = f(x_1^{(m)}, z_1^{(m)}, x_N^{(m)}, z_N^{(m)}, a^{(m)}, b^{(m)}). \quad (5)$$

Then the convergence criterion of this iterative calculation can be written as

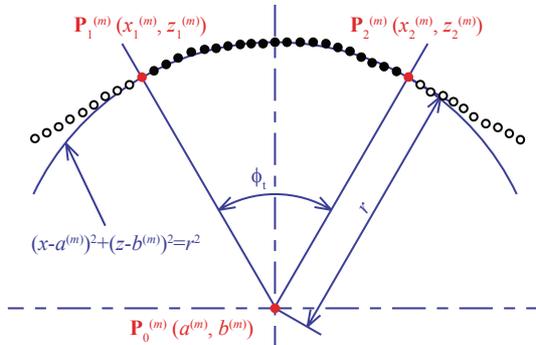


Fig. 5 Extraction of the dataset of the spherical part from the whole profile data corresponding to the central angle  $\phi$ .

$$\nabla F(\phi^{(m)})^T \cdot \mathbf{d}^{(m)} < 1 \quad (6)$$

where

$$\nabla F(\phi^{(m)}) = \begin{pmatrix} \partial F(\phi^{(m)}) / \partial x_1^{(m)} \\ \partial F(\phi^{(m)}) / \partial z_1^{(m)} \\ \vdots \\ \partial F(\phi^{(m)}) / \partial b^{(m)} \end{pmatrix} \quad (7)$$

and

$$\mathbf{d}^{(m)} = \begin{pmatrix} x_1^{(m)} - x_1^{(m-1)} \\ z_1^{(m)} - z_1^{(m-1)} \\ \vdots \\ b^{(m)} - b^{(m-1)} \end{pmatrix}. \quad (8)$$

Unfortunately, the function  $F(\phi^{(m)})$  is too complicated because it includes results of the least square analysis and it is not easy to show its convergence property with equations. In the analyses the author carried out, however, the iterative calculation converged in all cases. Fig. 6 shows the convergence property of the iterative calculation. Four cross sections were analysed with proposed method and in all cases, the central angle  $\phi^{(m)}$  converged to the target value  $\phi_t = 60^\circ$  with the iteration between 6 and 8. It suggests empirically that the algorithm satisfies its convergence criterion.

### 3.6 Analysis of the conical part

Once the blend points between the sphere and cone,  $\mathbf{P}_1$  and  $\mathbf{P}_2$ , are determined, the end points of verification of the generatrices,  $\mathbf{P}_3$  and  $\mathbf{P}_4$  can be determined as points 400  $\mu\text{m}$  distant from  $\mathbf{P}_1$  and  $\mathbf{P}_2$ , respectively. If the slope of the generatrices is assumed as  $30^\circ$  from  $x$ -axis, the coordinates of the end points can be expressed as

$$\mathbf{P}_3 (z_1 - 400 \mu\text{m} \cdot \cos 30^\circ, z_3), \quad (9)$$

$$\mathbf{P}_4 (z_2 + 400 \mu\text{m} \cdot \cos 30^\circ, z_4). \quad (10)$$

In those expressions,  $z_3$  and  $z_4$  should be picked from actual profile data.

The angles of the slopes of generatrices may be different from  $30^\circ$  in actual indenter. Even so, the maximum permissible error of this angle is  $0.17^\circ$  to  $0.35^\circ$ [16, 17] and it brings the error of the coordinates of the end points by only 0.17 % to 0.35 %. It is negligibly small.

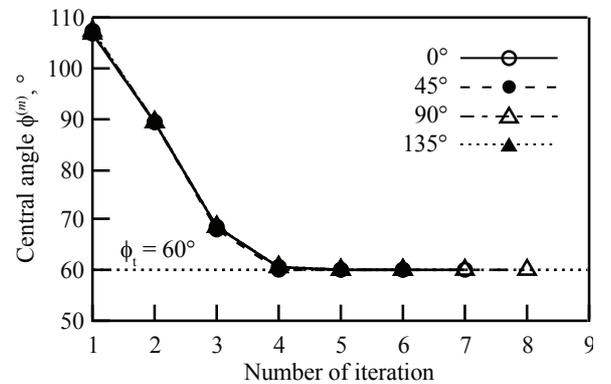


Fig. 6 Convergence property of the iterative calculation.

The included angle of the cone  $\alpha$  is calculated from two linear regression lines for the datasets picked from the ranges between  $P_1$  and  $P_3$ , and between  $P_2$  and  $P_4$ . When those regression lines are expressed as

$$z = p_1x + q_1 \quad (11)$$

and

$$z = p_2x + q_2 \quad (12)$$

respectively, the included angle  $\alpha$  is calculated with following equation,

$$\alpha = 180^\circ + \tan^{-1} p_1 - \tan^{-1} p_2 \quad (13)$$

The straightness of the generatrices can be evaluated as the deviation range from the regression lines, Eqs. (11) and (12), in the normal direction.

#### 4. RESULTS OF THE ANALYSIS AND DISCUSSIONS

In this paper, two kinds of analytical results are shown. One is the verification compliant to the specification of WGH or ISO. Another is a compatible method to the calibration method developed and actually used in NMIJ to establish the national standard indenters.

##### 4.1 WGH/ISO compliant verification

As an example, one cross section of a Rockwell diamond indenter was analyzed according to the procedure introduced in the previous section with the target central angle  $\phi_1 = 60^\circ$ . The center of curvature  $P_0$ , the blend points  $P_1$  and  $P_2$ , the end points for the evaluation of generatrices  $P_3$  and  $P_4$  are

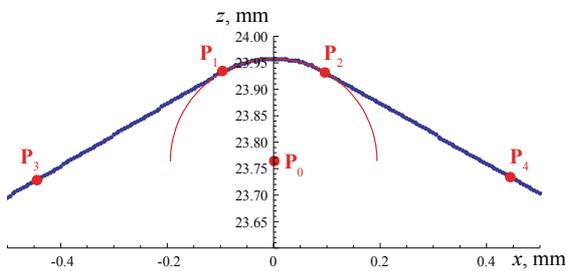


Fig 7 Graphical representation of the result.

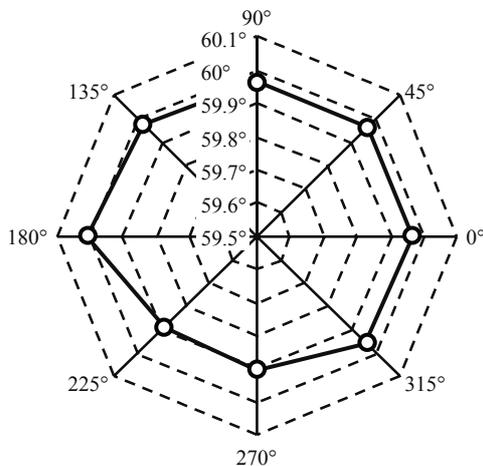


Fig 8 Angles of generatrices from the axis perpendicular to the datum plane in eight directions.

Table 2 Analytical results on single cross section.

Parameter	Proposed method	Simplified method
Cone angle, $\alpha_m$	119.855 °	119.851 °
Tip radius, $R_a$	194.120 $\mu\text{m}$	194.197 $\mu\text{m}$

illustrated with the fit circle on the profile data in Fig. 7. Calculated average tip radius  $R_a$  and the included angle of cone  $\alpha$  are also shown in Table 2.

In Table 2, the analytical results with proposed method are compared with the results with a simplified analysis. The simplified method is as follows. If the indenter has an ideal geometry specified by WGH/ISO, *i. e.*, 200  $\mu\text{m}$  of tip radius and 120° of included angle of cone, the blend points,  $P_1$  and  $P_2$ , locate at  $\pm 100 \mu\text{m}$  horizontally away from the axis of the indenter. So, as a simplified method, the dataset in this area could be used for the least square circle fitting. The table shows that there are no significant differences between two methods. The author thinks that the reason is the indenter under verification has good agreement to the specifications of ISO 6508. The error observed in this measurement is just a little bit more than the requirement in ISO 6508-3 and also the circularity of the cross section of spherical part seems to be good. If a variety of indenters are verified with this technique, more difference could be found.

For the evaluation of the axis of cone, the angles of generatrices from the axis perpendicular to the datum planes is illustrated in Fig. 8 in every 45°. It seems to be difficult to determine the direction of the cone axis because the measured geometry is not a perfect cone. This chart, however, shows that the angles around 135° to 180° are almost similar to the opposite side, *i. e.*, 315° to 0° whereas the angles around 45° to 90° can be recognized a little bit different from the angles on the opposite side, *i. e.* around 225° to 270°. It suggests that the axis of cone is declined approximately 0.03° in the direction around 225° to 270°.

##### 4.2 Compatibility with currently used verification/calibration method in NMIJ

In NMIJ, every standard indenter is characterized by the multiple regression analysis and has individual correction value to determine true hardness[3, 9]. The geometrical parameters used in this analysis are average tip radii in five different ranges set by an aperture in the microscopic measuring device and the cone angle. This aperture works to change effective numerical aperture (NA) of the optical system. Therefore, the range under evaluation is expressed by NAs (Fig. 9).

As seen in Fig. 9, the NA relates to the indentation depth  $h_c$ . Therefore it can be regarded that these parameters are equivalent to the relation between average radii of indenter tip with respect to different indentation depth. The relation between NA, central angle and contact depth is calculated for the indenter which has exactly shaped according to ISO standard are shown in Table 3. The range for NA = 0.65 is wider than the spherical part of ideally shaped indenter. The reason why the radius of the range beyond the blend point is included is to evaluate imperfection of the geometry around the blend point.

In order to keep the continuity between currently used verification results and newly introduced ones, it is useful

Table 3 Relation between the central angles and the contact depths of the ideally shaped indenter shown with the numerical apertures (NAs) of the optical measurement system of NMIJ.

NA	Central angle, °	Contact depth, $\mu\text{m}$
0.20	23.07	4.04
0.30	34.92	9.21
0.40	47.16	16.70
0.50	60.00	26.79
0.65	81.08	45.40 <sup>†</sup>

<sup>†</sup> including conical part of indenter

for us to express the geometry of spherical part of indenter as the function of central angle. The measuring method used in this investigation doesn't have the function to change the measuring range like the currently used instrument of NMIJ but the same parameters can be obtained if the iterative calculation described in previous section is used, *i. e.*, carrying out the iterative regression analysis with the target central angle  $\phi$  which corresponds to the NA of currently used verification instrument. The result of this technique is

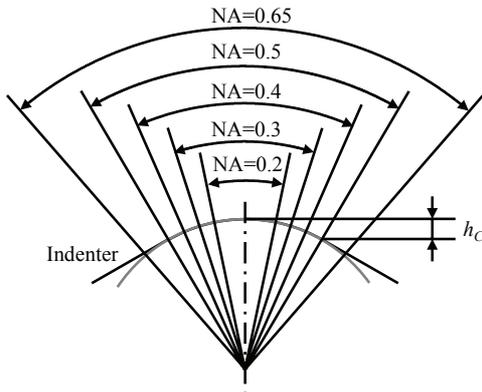


Fig. 9 Evaluation of geometry of spherical part of indenter adopted by NMIJ.

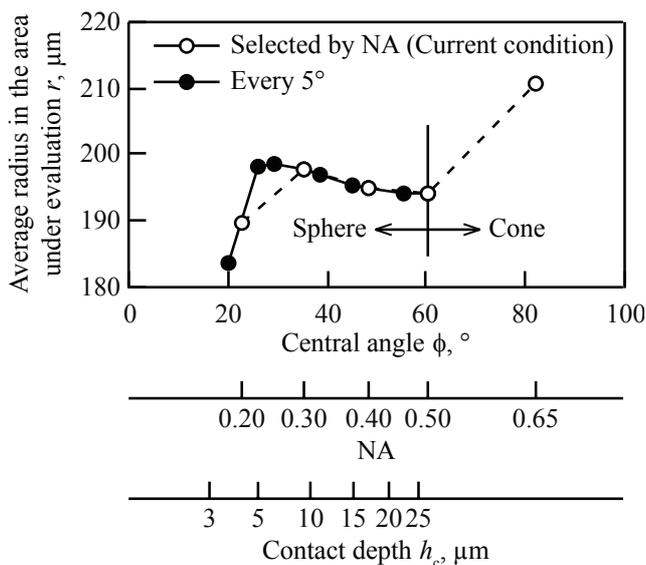


Fig. 10 Average tip radius in the different evaluation area on the spherical part of indenter.

shown in Fig. 10 as open dots. As seen in this chart, the average radius between NA = 0.20 to 0.50, *i. e.*, whole spherical part, is not constant for this indenter. In other words, the deviation from the definition ( $R_a=200 \mu\text{m}$ ) depends on the indentation depth. It suggests that the deviation of hardness of this indenter from true hardness would be different for different hardness level of test materials.

In Fig. 10, the average radii evaluated in every 5° of central angle are shown as solid dots to demonstrate the advantage of proposed analytical method. Those values are obtained from only one measurement but it is showing that the proposed method brings more information than currently used optical method. This result describes the geometry of this indenter in more detail, *i. e.*, the radius of this indenter is almost constant in most of the range but smaller just vicinity of the tip.

## 5. CONCLUSIONS

The geometry of a Rockwell diamond indenter was measured with a laser probe 3D profile measurement instrument. The result was analysed according to the definition by CIPM/CCM/WGH and the method compatible to the one currently employed in NMIJ for the national standard indenters. In order to carry out those verifications, the iterative regression method is proposed to determine desirable range for the verification. It helps us to determine exact positions of the blend of sphere and cone or specified range necessary for the characterization of indenters. Results of analysis of a Rockwell diamond indenter are shown as an example for the both purposes.

The example demonstrates that the method can be determine exact blend points of cone and sphere and it enables us to verify indenters fully compliant way to the definition of WGH.

It is also shown that the same result with currently used method in NMIJ can be obtained even though different principles and instrument are used for current and newly proposed procedures. Newly proposed method suggests the capability to describe the geometry in more detail than currently used method. It is expected that the characterization of standard indenters, *i. e.*, specific bias of each standard indenter from the true hardness value, will be carried out in better accuracy and minimizing the uncertainty of the national standard indenters.

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