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Direct Verification and Calibration of Rockwell Diamond Cone Indenters

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Abstract – The direct verification and the calibration of Rockwell diamond cone indenters in NMIJ and JBI are described. Developed instrument can verify the roundness of the spherical part of the indenters, measure the radius of indenter tip and the cone angle by the optical methods. The regression analysis of the geometry of indenters and hardness value can determine the bias of each indenter in each hardness level. This bias can be used to correct the hardness value. To investigate the availability of the correction of hardness value, the experiments were carried out to analyze the relation between the indenters and other testing conditions. The results show that the effect of indenters are independent with other factors and the correction of hardness values are available. Hardness values were agreed within 0.04 HRC (standard deviation) after the correction is was applied.

Keywords: Rockwell diamond cone indenter, Multiple regression, Analysis of variance

1. INTRODUCTION

An indenter is one of the important parts in a hardness testing machine. Because the testing force is applied through this part, the performance of the indenter exerts significant effect on the hardness value. Especially for the Rockwell hardness, it is well-known that the some scales of Rockwell hardness which uses a diamond cone indenters is strongly influenced by the performance of indenters. The shape of the indenter is a cone with a small curvature on the apex. This complicated geometry makes it difficult to realize exact shape. In addition, a single crystal of diamond is difficult to shape into a sphere because of its anisotropy.

The verification of indenters is required in ISO standards [1, 2]. However the detail of the method is not clearly specified in this standard and actually there are some kinds of verification methods are used in the national metrology institutes (NMIs) or the calibration laboratories. NMIJ and JBI were developed the method of the verification and calibration of Rockwell diamond cone indenters [3] and it is now already in service. This method is also different from any other methods and has some different characteristics.

In this paper, the method to the direct verification and calibration of Rockwell diamond cone indenters are presented. The bias of the hardness values is also discussed.

2. DIRECT VERIFICATION OF INDENTERS

The geometry of Rockwell diamond indenters should be verified according to ISO 6508-2 and 3 [1, 2]. The items to be verified are the cone angle, the radius of the indenter tip, etc and usually these items are measured with surface profilers or profile projectors. In many cases, results of the measurement are expressed with two parameters, *i. e.*, the tip radius and the cone angle. However it is not enough to describe the characteristics of the indenter shape because the contact area of the indenter can be changed with the indentation depth (therefore with the hardness value). It is better to verify the indenter shape considering its indentation depth. Actually, the characteristics of the indenters used for Martens hardness are expressed as the function of the indentation depth (or contact depth).

2.1 Description of the instrument

NMIJ and JBI developed an improved method to measure the geometry of indenters [3]. In this method, six parameters are used to describe the geometry of indenters, *i. e.*, five parameters are used to express the radius of indenter tip and one parameter represents the cone angle. These parameters are measured with an optical device specially designed for the verification of Rockwell diamond indenters. The appearance of this instrument is shown in Fig. 1. This instrument consists of an indenter holder on the moving head, a stabilized laser as a light source, an optical unit and a rotary encoder. There are three functions with this instrument, *i. e.*, the verification of the roundness, the measurement of the radius and the measurement of the cone angle. An operator can change the layout of the optical unit with some adjusting levers to use each function.

2.2 Verification of the roundness

The first function of this instrument is to verify the roundness of the spherical part of an indenter. The optical layout for this function is shown in Fig. 2. This optical system works as an interference microscope. A precise small sphere (bearing ball) is used as a reference surface and the interference between the reflections from the indenter tip and the reference makes an image shown in Fig. 3. The rings in these images represent the roundness of the indenter tip

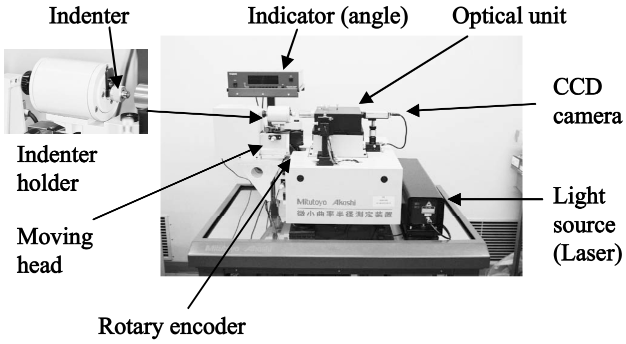


Fig. 1. Measuring instrument for Rockwell diamond indenters (Mitutoyo/Akashi)

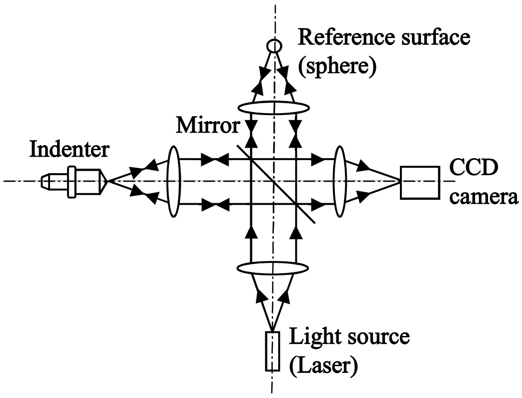


Fig. 2. Optical layout for the verification of the roundness

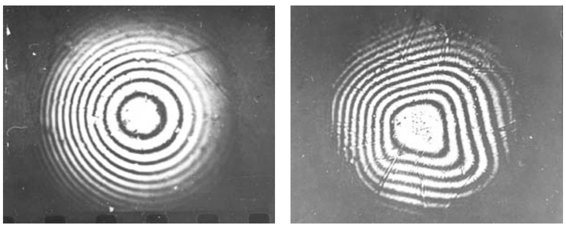

(a) Good roundness (b) Bad roundness

Fig. 3. Examples of observed images in the verification of the roundness.

and it provides some information to classify the grades of indenters.

2.3 Measurement of the radius

The second function of this instrument is to measure the radius of the indenter tip. The optical layout for this function is shown in Fig. 4. It works as a microscopic collimator. The light induced through a pinhole is focused on the indenter. The position of the indenter can be adjusted along with the axis of the light. When the image of the pinhole is focused on the surface of the indenter tip, the image of the pinhole can be observed by the CCD camera (The intensity of the light is also evaluated by a photo-detector). As well as this alignment, when it focused on the center of the spherical part of the indenter, the same image of the pinhole can be also observed. In this procedure, the difference between those two positions represents the average radius of the indenter tip. The variation of observed images and the light intensity are shown in Fig. 5. Area of the spherical part can be adjusted by an aperture. The relation between numerical apertures (NAs) and the area to be verified is shown in Fig. 6 and Table 1. As shown in this table, the area for $NA = 0.65$ exceeds the border between the spherical part and the conical part (Center angle of 60.00°, $h_C = 26.79\,\mu$m). If the shape of the indenter is correct, this condition of NA is not necessary. However, this border is not clear in actual

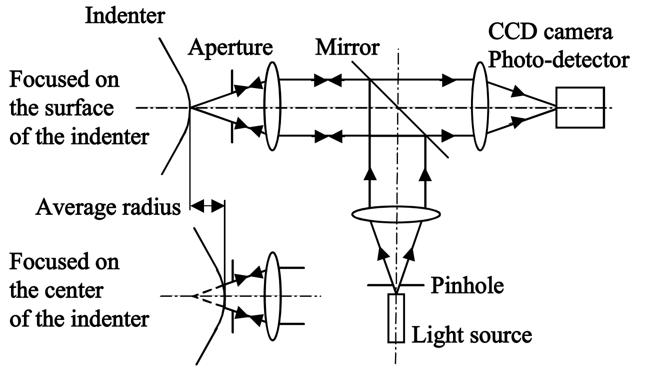
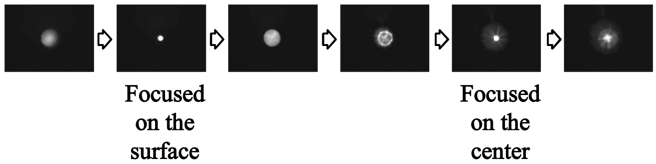
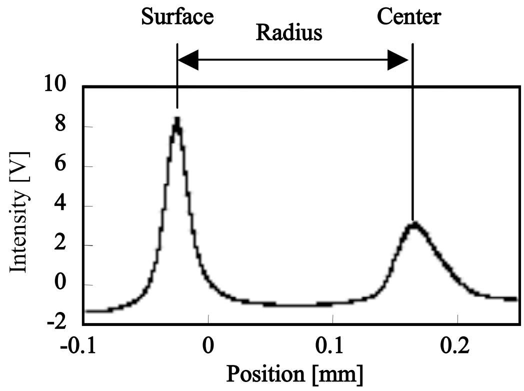


Fig. 4. Optical layout for the measurement of the radius



(a) Image of the reflection



(b) Intensity of the reflection

Fig. 5. An example of the measurement of the radius

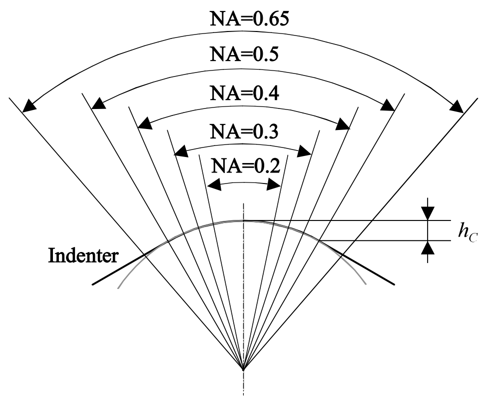


Fig. 6. Relation between the area to be measured and the NA of the objective.

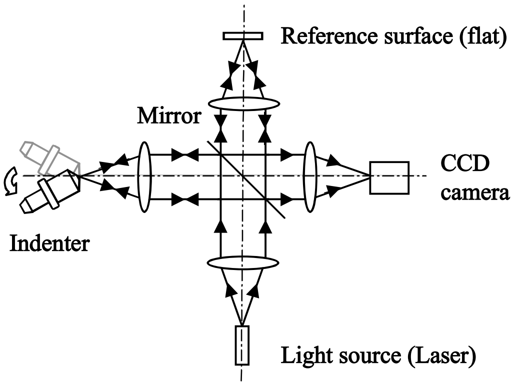


Fig. 7. Optical layout for the measurement of the cone angle.

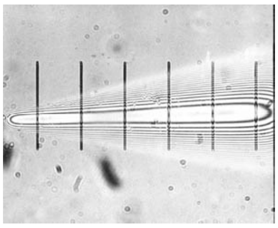


Fig. 8. An example of observed image in the measurement of cone angle.

indenter and it has still some curvature in many cases. In this method, it is included to take into account this effect.

TABLE 1. The relation between NAs and the area to be verified.

NA	Center angle [°]	Indentation depth * [μm]
0.2	23.07	4.04
0.3	34.92	9.21
0.4	47.16	16.70
0.5	60.00	26.79
0.65	81.08	---

* Indentation depths are calculated assuming the perfect sphere shape.

2.4 Measurement of the cone angle

The third function of this instrument is to measure the cone angle. The optical layout for this function is shown in Fig. 7. It works as an interference microscope. The difference from Fig. 2 is the direction of the indenter and the flat reference surface. When the indenter is placed so that the generatrix of the cone is perpendicular to the light, some pattern around the generatrix can be observed (Fig. 8). It means that this image can be used as a guide to adjust the direction of the indenter. The directions of the indenter are measured with a rotary encoder in the both side of the indenter and the cone angle is calculated from the angle of rotation of the indenter holder.

2.5 Examples of the verification results

Examples of the verification results are shown in Table 2. Usually these results are reported with the estimation of uncertainty. Values of radius are the average value in the area to be verified. If it is required to use only one value, the value for the maximum NA is available because it represents average characteristics of the hole part of the sphere.

TABLE 2. Verification results

		P1	P2	P3
Radius ρ, μm	NA = 0.2	197.5	188.9	217.0
	NA = 0.3	199.1	186.9	199.3
	NA = 0.4	197.6	193.5	196.4
	NA = 0.5	198.9	192.7	197.5
	NA = 0.65	199.6	194.1	196.8
Cone angle, °		120.11	119.99	120.17

3. CALIBRATION OF INDENTERS

The geometric error of the indenter causes the change of the indentation depth. It means that each indenter has its own bias of hardness value. In this section, the method to determine the bias of each indenter is described. The unstable fixture of the diamond tip also causes the change of hardness value and it is usually verified by the manufacturer in the indirect method. In this paper, it is assumed that all indenters have passed this verification and the effect of the fixture of the diamond tip is negligible. Therefore it can be considered that the bias depends on only its geometry.

3.1 Additivity of the hardness value

In general, there is no additivity for the hardness values. It means that no one can add or substitute hardness values and it also means that the hardness values cannot be corrected. In practice, it seems however to be reasonable to use the bias of the indenter to correct hardness value because it is clear that there is a relation between the indenter shape and hardness value. To investigate the possibility of the correction of hardness value, measurements of the hardness blocks were carried out in systematically designed conditions and the relation between hardness value and some testing conditions are analyzed. If the result shows that the bias of an indenter is not changing by other testing conditions, it can be concluded that the effect of indenter is independent of other factors and the correction of hardness value is available.

As the factors of testing condition, reference blocks (hardness values), indenters, the indentation speed and the holding time of the total testing force were considered for this experiment (Table 3). Geometry of the indenters (P1, P2 and P3) were shown in Table 2. Some conditions which do not conform with ISO standard were chosen for this experiment to investigate the difference from the traditional condition in Japan. Measurements were carried out randomly and the analysis of variance (ANOVA) was carried out for each hardness level.

Fig. 9-11 show the main effects of four factors at three hardness levels. The effect of indenters are similar in those three hardness levels. It can be understood because the radius of these indenters are not so changing with NAs (Table 2) and the characteristics of these indenters seem not to changing. Effects of the indentation speed and the holding time of the total test force are similar to the previous study [4].

The results of ANOVA are shown in Table 4-6. At 40 and 60 HRC levels, some factors of interactions are seems to be significant (the mark of "*" means "significant in 95% confidence level" and the mark of "**" means "significant

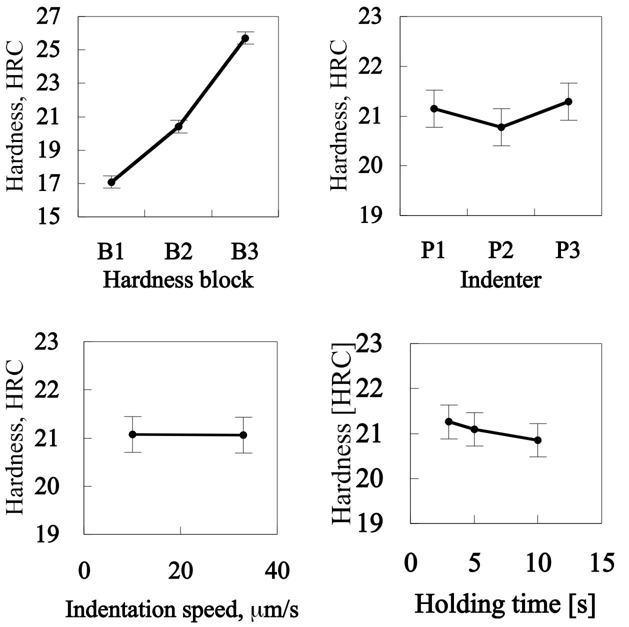


Fig. 9. Main effects of factors at 20 HRC level.

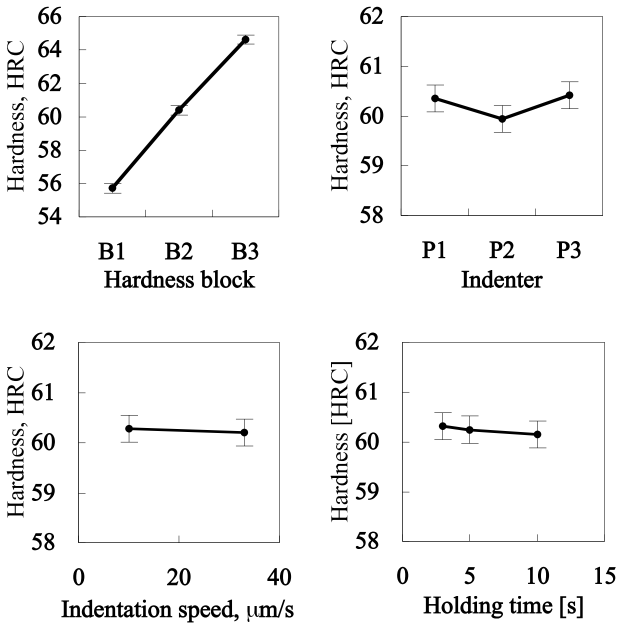


Fig. 11. Main effects of factors at 60 HRC level.

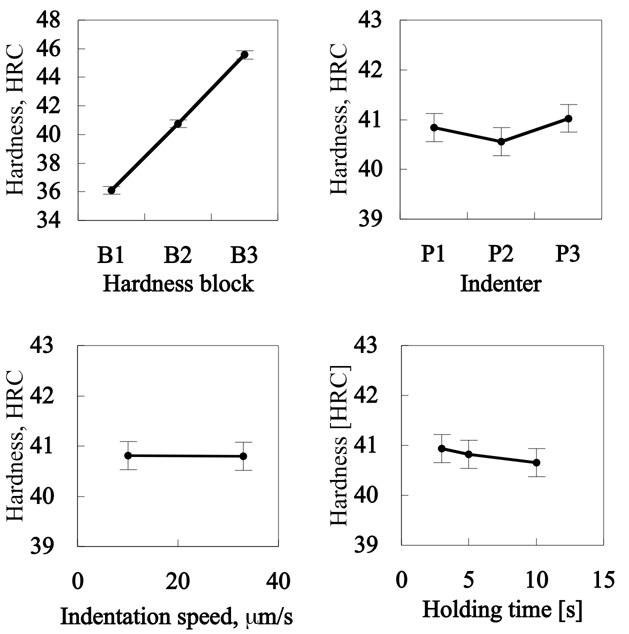


Fig. 10. Main effects of factors at 40 HRC level.

in 99% confidence level"). If $\mathrm{B} \times \mathrm{P}$ is significant, it means that the effect of indenter is different in each hardness, *i. e.*, in each indentation depth. It is consistent with the assumption described above. Other significant factors are related with the indentation speed or the holding time of the total testing force. They can be understood as the effect of creeping.

In these results, no significant interactions between indenters and other factors (except the interaction with hardness block). It can be concluded that the effect of indenter is independent from other testing conditions and the hardness values can be corrected.

TABLE 3. Factors and their levels

Factor	Symbol	Level
Hardness block *	B	17.1, 20.4, 25.7 [HRC] (for 20 HRC) 36.1, 40.8, 45.6 [HRC] (for 40 HRC) 55.7, 60.4, 64.6 [HRC] (for 60 HRC)
Indenter	P	P1, P2, P3
Indentation speed	V	10, 33 [μm/s]
Holding time of total test force	D	3, 5, 10 [s]

* Three levels with small difference of hardness value are considered in each hardness level.

3.2 Method to determine the bias

The bias of the hardness value for an indenter is expressed as

$$\Delta H = H_i - H_0, \quad (1)$$

where H_i is the hardness value for the indenter and H_0 is the hardness value for the ideal indenter. The ideal indenter is the indenter which has a exact shape specified in ISO standard, *i. e.*, 200 μm of tip radius and 120° of the cone angle. Actually the value of H_0 is unknown but it can be estimated by a statistical method.

To define the bias of the indenters, it is required to prepare a number of indenters because the statistical analysis requires enough number of the population. The relation between the hardness and the parameters of geometry is assumed as

$$H_i - H_1 = \sum_{j=1}^{5} \beta_j(\rho_{ij} - \rho_{1j}) + \beta_6(\theta_i - \theta_1) \quad (2)$$

where $\rho_{i1}, \rho_{i2}, \cdots, \rho_{i5}$ are radii of indenter *i* measured with the microscopic collimator in different apertures. θ_i is the cone angle of indenter *i*. H_1, ρ_{1j} and θ_1 are the hardness, radii and the cone angle of one of the indenters (reference

TABLE 4. Analysis of variance for 20 HRC level. (4-way layout with 4 population parameters)

Source	Symbol	Sum of squares, S	DOF f	Variance, V	Variance ratio, F_O	Pooled V	DOF f'	F_O'
Hardness block	B	4088.0184	2	2044.0092	146656.99 **		2	14655.22 **
Indenter	P	15.2342	2	7.6171	546.52 **		2	56.61 **
Indentation speed	V	0.0099	1	0.0099	0.71		1	0.07
Holding time	D	8.9809	2	4.4905	322.19 **		2	32.20 **
Interaction	B × P	0.0875	4	0.0219	1.57			
	B × V	0.0029	2	0.0014	0.10			
	B × D	0.0837	4	0.0209	1.50			
	P × V	0.0275	2	0.0137	0.99			
	P × D	0.0661	4	0.0165	1.19			
	V × D	0.0244	2	0.0122	0.88			
	B × P × V	0.0311	4	0.0078	0.56			
	B × P × D	0.0505	8	0.0063	0.45			
	B × V × D	0.0117	4	0.0029	0.21			
	P × V × D	0.0571	4	0.0143	1.02			
	B × P × V × D	0.0603	8	0.0075	0.54			
Error	e	3.7631	270	0.0139		0.1395	316	
Total	T	4116.5093	323					

TABLE 5. Analysis of variance for 40 HRC level. (4-way layout with 4 population parameters)

Source	Symbol	Sum of squares, S	DOF f	Variance, V	Variance ratio, F_O	Pooled V	DOF f'	F_O'
Hardness block	B	4835.0897	2	2417.5448	289462.13 **		2	30995.39 **
Indenter	P	11.9901	2	5.9950	717.81 **		2	76.86 **
Indentation speed	V	0.0052	1	0.0052	0.62		1	0.07
Holding time	D	4.3641	2	2.1821	261.27 **		2	27.98 **
Interaction	B × P	0.0238	4	0.0060	0.71			
	B × V	0.0223	2	0.0111	1.33			
	B × D	0.1175	4	0.0294	3.52 **			
	P × V	0.0041	2	0.0021	0.25			
	P × D	0.0294	4	0.0073	0.88			
	V × D	0.0067	2	0.0034	0.40			
	B × P × V	0.0020	4	0.0005	0.06			
	B × P × D	0.0340	8	0.0042	0.51			
	B × V × D	0.0005	4	0.0001	0.01			
	P × V × D	0.0075	4	0.0019	0.23			
	B × P × V × D	0.0291	8	0.0036	0.44			
Error	e	2.2550	270	0.0084		0.0780	316	
Total	T	4853.9811	323					

TABLE 6. Analysis of variance for 60 HRC level. (4-way layout with 4 population parameters)

Source	Symbol	Sum of squares, S	DOF f	Variance, V	Variance ratio, F_O	Pooled V	DOF f'	F_O'
Hardness block	B	4296.7369	2	2148.3684	584933.92 **		2	29569.73 **
Indenter	P	14.0017	2	7.0008	1906.11 **		2	96.36 **
Indentation speed	V	0.4225	1	0.4225	115.03 **		1	5.82 *
Holding time	D	1.4672	2	0.7336	199.74 **		2	10.10 **
Interaction	B × P	0.0709	4	0.0177	4.83 **			
	B × V	0.0617	2	0.0308	8.39 **			
	B × D	0.0148	4	0.0037	1.01			
	P × V	0.0013	2	0.0006	0.18			
	P × D	0.0083	4	0.0021	0.57			
	V × D	0.0017	2	0.0008	0.23			
	B × P × V	0.0009	4	0.0002	0.06			
	B × P × D	0.0407	8	0.0051	1.39			
	B × V × D	0.0133	4	0.0033	0.91			
	P × V × D	0.0131	4	0.0033	0.89			
	B × P × V × D	0.0096	8	0.0012	0.33			
Error	e	0.9917	270	0.0037		0.0727	316	
Total	T	4313.8564	323					

indenter). $\beta_1, \beta_2, \cdots, \beta_6$ can be obtained through the regression analysis. When the coefficients β_j are determined, the hardness for the ideal indenter is obtained substituting the specified values to the geometrical parameters,

$$H_0 - H_1 = \sum_{j=1}^{5} \beta_j (200 - \rho_{1j}) + \beta_6 (120 - \theta_1) \quad (3)$$

In the regression analysis, there comes some residual errors. It should be considered as the uncertainty of the estimated bias.

The biases of the indenters used for the experiment is shown in Table 7 (Some indenters are chosen for this experiment because they has large numbers of the biases). The bias is determined in every hardness level. It can be realized because the regression analyses were carried out considering the detailed geometry with six parameters. After the correction, the difference of hardness values is typically 0.04 HRC (standard deviation).

TABLE 7. The biases of the indenters.

Hardness level	P1	P2	P3
20	0.06	-0.38	0.21
25	0.05	-0.25	0.19
35	-0.04	-0.29	0.27
40	-0.04	-0.29	0.27
45	-0.02	-0.29	0.12
55	0.07	-0.34	0.08
60	0.07	-0.34	0.08
65	-0.04	-0.43	0.08

4. CONCLUSION

In this paper, the direct verification and the calibration of Rockwell diamond cone indenters in NMIJ and JBI are described. To investigate the availability of the correction of hardness value, the experiments were carried out to analyze the relation between the indenters and other testing conditions. The results show that the effect of indenters are independent with other factors and the correction of hardness values are available. Hardness values were agreed within 0.04 HRC (standard deviation) after the correction is was applied.

To show the compatibility with other calibration laboratories, it is expected to compare the results with other verification methods.

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