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WIDE-RANGE VERIFICATION OF THE GEOMETRY OF VICKERS DIAMOND INDENTERS

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Abstract: The three different methods, the NRLM-type Vickers indenter verification instrument, the scanning white light interferometry and the atomic force microscope, are used for the wide-range verification of a Vickers indenter. Those methods are available in different range of indenter surface and it was found that the integration of different instruments are helpful for the better understanding of the thorough geometry of a Vickers indenter. In addition, an improved instrument is developed for low-force and microhardness range and evaluated its availability. The results show that it can indicate the similar value to the most reliable instrument even in the smaller range of indenter and it suggests that the range of the reliable verification may be expanded to low-force and microhardness.

Keywords: direct verification, scanning white light interferometry, AFM.

1. INTRODUCTION

The “hardness test” is the evaluation method for the strength of materials by measuring the deformation on the specimen surface, which is created by pressing an indenter into the material with a definite testing force. There are several scales of hardness used in the industries. The difference between those scales is mainly about types of indenter and the measuring method of indentation. In case of Vickers hardness, a tetrahedral diamond tip is used as an indenter. The advantage of this type of indenter is that a geometrically similar indentation can be made with any levels of testing force. Typical range of testing force is from less than 0.1 N to 980.7 N. Because of this advantage, Vickers hardness is used especially with low testing forces, *i. e.*, Vickers microhardness or nano-indentation for the evaluation of small parts of materials or thin films, in these days.

The ISO standard [1-3] is requiring the verification of hardness machines and it includes the direct verification of indenter geometry. For Vickers indenters, it is necessary to consider the range of indenter tip in which the indenter geometry is measured. Typical ranges under various testing forces are shown in Table 1. If this indenter are used for the microhardness, the geometry of it should be measured in the small area around the tip, such like 20 or 30 µm.

Several methods are being used for the verification of one Vickers indenter at the calibration laboratories. Among them, the interference microscope is one of the most reliable method for direct verification of the geometry of Vickers indenters. With this method, an indenter is held with an indenter holder which is mounted on the rotation table. The normal direction of the face of pyramid is determined by the fringes on the observed surface. When a flat surface is observed with a interference microscope, a pattern of fringes can be observed according to the misalignment of the observed surface to the optical path. If no fringes can be observed by adjusting the direction of the indenter, the direction of the face of pyramid is determined and it can be measured with rotary encoders equipped on the rotation table.

This method is quite effective to the precise verification of Vickers indenter geometry and in fact some national laboratories are using this kind of instrument. However, the angle sensitivity of this method is getting lower when the smaller area of a face is measured. It means that this method is not always suitable for the verification of Vickers indenter for low-force or microhardness.

In this study, several instruments were used to measure the indenter geometry and the availability of them to the Vickers indenter verification for various ranges was discussed. In addition, an attempt to improve the measurement capability of Vickers indenter geometry in low-force and microhardness ranges was reported.

Table 1. Typical ranges of the contact surface of a Vickers indenter under various testing forces.

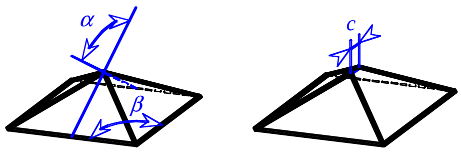
Designation	Testing force*, N	Indentation depth**, µm	Diagonal length**, µm
Hardness test	49.03 – 980.7	14.50 – 137.5	101.5 – 962.8
Low-force hardness test	1.961 – 29.42	2.900 – 23.82	20.30 – 166.8
Microhardness test	0.9807 – 0.09807	2.857*** – 4.350	20.00*** – 30.45
Nano-indentation	Not specified in ISO 14577-1		

* Recommendation in ISO 6507-1
** Hardness range is assumed as 200 to 900 HV
*** Lower limit of diagonal length in ISO 6507-1

2. VERIFICATION ITEMS OF VICKERS INDENTER

The geometry of the Vickers pyramid is defined with the angle between opposite faces of 136 degrees. To ensure the accuracy of its geometry, ISO 6507-3 requires to verify following items (See Fig. 1);

a) The flatness of four faces shall be within 0.0003 mm,
b) The angle between the opposite faces (α) shall be 136 ± 0.1º,
c) The angle between the axis of the diamond pyramid and the axis of the indenter-holder shall be less than 0.3º,
d) The line of conjunction between opposite faces (c) shall be smaller than 0.001 mm, 0.0005 mm and 0.00025 mm for the range of testing force of $F \geq$ 49.03 N, 1.961 N $\leq F <$ 49.03 N, and 0.09807 N $\leq F$ 1.961 N respectively.
e) The corner angle of the square base of pyramid (β) shall be 90 ± 0.2º.



(a) (b)

Fig. 1. Geometrical parameters (verification items) of Vickers indenter

(a) The angle between the opposite faces (α) and the corner angle of the square base of pyramid (β).
(b) The line of conjunction between opposite faces (c).

Sometimes the geometry of Vickers pyramid is verified by measuring the angle between opposite edges as noted in ISO 6507-2 and it shall be 148.11º. Strictly speaking, this alternative method, however, cannot show that the angle between opposite faces is also true. So it is better to measure the normal direction of four faces and evaluate the angle between opposite faces directly.

It should be considered that the several verification items, *i. e.*, the items c) and e) of previous list, can be affected by the disagreement between;

a) The coordinates of the pyramid,
b) The coordinates of the indenter holder,
c) The coordinates of the measuring devices.

In general, these coordinates cannot be separated from measurement results. In this paper, the coordinate of the pyramid is defined as follows.

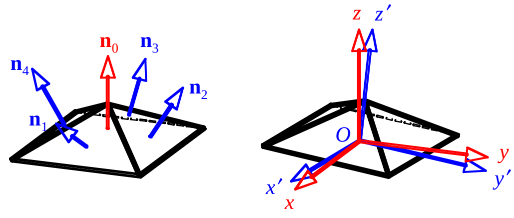
Let $\mathbf{n}_1$, $\mathbf{n}_2$, $\mathbf{n}_3$ and $\mathbf{n}_4$ are the unit normal vectors of four faces in the coordinates of the measuring device (See Fig. 2). The axis of the pyramid can be determined as,

$$\mathbf{n}_0 = \frac{1}{l}\sum_{i=1}^{4}\mathbf{n}_i \qquad (1)$$

where

$$l = \left|\sum_{i=1}^{4}\mathbf{n}_i\right| \qquad (2)$$

The correction of coordinates has a significant effect not only the verification of geometry but on the calculation of the area function for the instrumented indentation test.



(a) (b)

Fig. 2. Coordinates of a Vickers indenter

(a) Definition of the coordinates of pyramid.
(b) Difference of coordinates between the pyramid ($O-x'y'z'$) and the measuring instrument ($O-xyz$).

3. AVAILABILITY OF VARIOUS MEASURING INSTRUMENTS

Three different instruments were used to measure the indenter geometry to discuss their characteristics. The indenter under measurement is manufactured by Tokyo Diamond Tool MFG. Co. Ltd. and it was supplied for the Mitutoyo (former Akashi) Vickers hardness machine.

3.1. NRLM-Type Vickers Indenter Verification Instrument (VIVI)

Although the geometry of Vickers indenter is defined by the angle between opposite faces of pyramid, it is verified by measuring the angle between edges in many cases. The reason is that the angle between faces is not easy to measure and it may bring wrong results of verification. This instrument was developed by NLRM (former NMIJ) and it is currently used by JBI to provide their calibration service of Vickers indenters [4]. The measuring principle is similar to an autocollimator (Fig. 3). An image of a reticle is projected on the indenter surface and it is observed through an ocular to find the normal direction of a face. The direction of the normal of the face is measured with the dials on rotating tables. When 20x objective lens is used, the center of field of view is the point 150 μm distant form the indenter tip. This instrument can measure at only one point at once but if the flatness of the faces is acceptable, the indenter geometry can be verified in the range for normal Vickers hardness. The measurement uncertainty of this method is 2 minutes for the face angle and 4 minutes for angles of square base of pyramid (k = 2). This instrument can be calibrated with angle gauge blocks of ±5 seconds of accuracy.

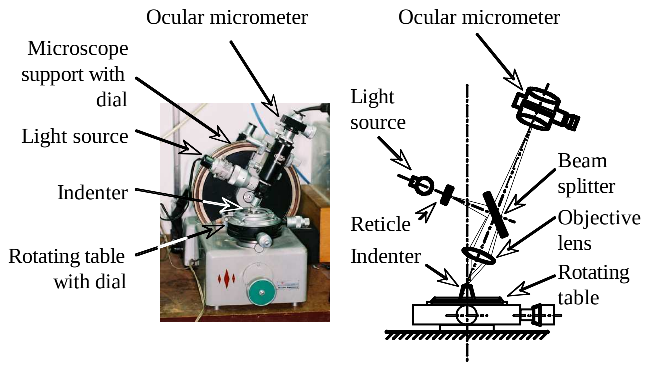


Fig. 3. NRLM-type Vickers indenter verification instrument

3.2. Scanning White Light Interferometry (SWLI)

There are several types of instruments which can measure three dimensional image of a body with an optical microscope, *e. g*. a scanning laser conforcal microscope. In this study, Zygo NewView 5000 system, which is based on scanning white light interferometry, were used. When a Vickers indenter is verified with an optical microscope, normally it is necessary to use an objective lens of 50x or higher because of its numerical aperture (the maximum slope with 50x objective is 24.27º, while the slope of Vickers pyramid face is 22º from the horizontal plane). When a 50x objective is used, the viewing area is approximately 0.14x0.11 mm with the resolution of 0.64 µm in horizontal and 0.1 nm in vertical. Therefore its application for indenter verification is ranging from low-force hardness to microhardness.

This instrument was calibrated with the standard samples (VLSI SHS-1.8QC and Zygo Lateral Calibration Standard 6300-2198-01) and horizontal pitch of 10 µm and step height of 1.828 µm was used (The expanded uncertainties are 0.018 µm and 0.011 µm, respectively).

3.3. Atomic Force Microscopy (AFM)

In this study, Pacific Nanotechnology Nano-R system was used. In general, a piezo-electric scanner of AFM has some nonlinear behavior and it can be the most significant error factor of measurement. The system used for this study includes a closed-loop control of a scanner to improve the linearity. The scanning range of this instrument is 80 µm with the resolution of 2 nm or better in the horizontal directions and the linearity of 1 % or better (with a closed-loop operation). In the vertical direction, moving range is 8 µm with the noise level of 0.13 nm RMS or better (closed-loop operation). Because a Vickers indenter has big difference of height from the tip to the edge of viewing area, the scanning area is limited and it is typically less than 15 µm. For this reason, the application of this instrument is limited to nano-indentation. If the rounding of tip or the truncation depth have to be verified, the measuring range should be chosen as a suitable magnification. In this study, the range of 5.4 µm was used.

The coordinates of the AFM is calibrated with the standard sample (VLSI STS3-1800P) and the horizontal pitch of 10.00 µm and the step height of 181.1 nm were used. The expanded uncertainties are 0.03 µm and 2.0 nm, respectively.

3.4. Results and Discussion

The result of VIVI is shown in Table 2. Among those three instruments, it can measure the angles of indenter with the smallest uncertainty. It is found that the reproducibility of measurement can be improved after considering the disagreement of axes of the indenter and the table.

Table 2. Results of VIVI.

Face angles	Position	Face 1 - 3		Face 2 – 4	
	Value	136°06′		136°04′	
Inclination*		04′			
Corner angles**	Position	Face 1-2	Face 2-3	Face 3-4	Face4-1
	Value	90°01′	89°58′	90°08′	89°53′

* Inclination of the axis of the diamond pyramid to the axis of indneter base plane (disagreement of the coordinates)

** Angle of the quadrilateral which formed by the intersection of the faces with a plane perpendicular to the axis of pyramid

The result of SWLI is shown in Fig. 4 (left). This instrument showed the good stability of measurement and the most significant source of uncertainty seemed to be the uncertainty of the reference standard. It is found that the observed image itself is quite fine and it can be a practical tool of indenter verification.

The result of AFM is shown in Fig. 4 (right). For this instrument, the most significant source of uncertainty is also the reference standard. The relative uncertainty is getting greater when the smaller area is observed and the uncertainty of angle measurement is also getting worse. In addition, this method is a contact method and it should be considered that the shape of cantilever tip may affect the measurement results. The vibration during the measurement makes some influence to the fineness of the image and the careful operation is always required for this measurement.

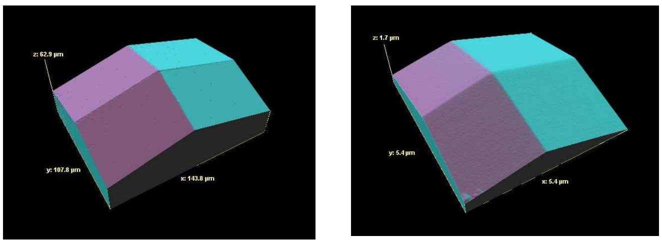


Fig. 4. Obtained images by SWLI (left) and AFM (right)

The face angles (α) and the corner angles of the square base of pyramid (β) are calculated from the images of SWLI and AFM. The results were compared with the results of VIVI in Table 3. It should be noted that those results are obtained with the same indenter but the location to determine the angles are different according to the magnification of the microscope. Therefore even if there are no errors with those measurements, the values may not exactly agree each other among those three instruments.

The results show that the values of VIVI and SWLI are close to each other but the values of AFM is different from other two instruments. The reason of this can be assumed that the stability of the instrument itself is getting worse with the magnification and also the uncertainty of the reference standards is getting greater and it may increase the error of the measurement results. The reference standards used to calibrate those three instruments are summarized in Table 4. While the accuracy of the angle gauge block is fairly good, the height standard for AFM has poor in the percentage uncertainty. For VIVI, there are fewer error sources than any other instruments because it can measure the direction of faces directly and also it can be calibrated with angle standard directly. Then it could be concluded that VIVI is regarded as the most reliable instruments among those verification methods. The difference of SWLI and AFM from VIVI may suggest that it is not easy to measure the angle of 0.1°, which is required value for the hardness calibration machine [3]. However, those three methods are used in the different range and the instrument for verification should be chosen in the same range of use of indenter.

Table 3. Comparison between three instruments.

Item	Position	VIVI	SWLI	AFM
α	Faces 1 – 3	136.10°	136.12°	137.36°
	Faces 2 – 4	136.07°	136.07°	137.07°
	Average	136.08°	136.09°	136.21°
β	Faces 1 – 2	90.02°	90.11°	89.32°
	Faces 2 – 3	89.97°	89.57°	91.53°
	Faces 3 – 4	90.13°	90.40°	89.27°
	Faces 4 – 1	89.88°	89.91°	89.88°
	Range	0.25°	0.83°	2.21°

Table 4. Reference standards used to calibrate the verification instruments.

Instruments	VIVI	SWLI	AFM
Reference standard	Angle gauge block	Standard sample	
Uncertainty (or accuracy)	± 0.0014°	Pitch: 10.008 ± 0.018 μm Height: 1.828 ± 0.011 μm (k = 2)	Pitch: 2.99 ± 0.02 μm Height: 181.6 ± 2.0 nm (k = 2)

4. DEVELOPMENT OF IMPROVED INSTRUMENT

The discussion in the previous section shows that VIVI has the best resolution and measurement uncertainty of the angle measurement. However, It cannot be applied to the verification of a low-force or microhardness indenter because of its low magnification. On the other hand, SWLI and AFM have enough magnification and resolution to observe a microhardness indenter but the measurement uncertainties of them are not enough for the verification of indenter geometry especially for the hardness calibration machines.

In order to establish the verification method for low-force and microhardness indenters, an improved measuring instrument is developed to evaluate the availability of the measuring method. The principle of this instrument is similar to VIVI. The light is induced to the face of pyramid and the normal direction of it is detected with the reflection of the light.

The higher magnification of objective lenses are applied to observe the smaller area around the indenter tip and the narrow beam of the light is used for the fine locating of the measuring point of the pyramid face. It is expected that these improvement enables to verify low-force or microhardness indenters.

The layout of the instrument is shown in Fig. 6.

An indenter is fixed on the indenter holder which is mounted on the five-axes stage. XYZ axes are used to adjust the position of the indenter and two rotational axes are used to adjust the direction of the indenter. Each rotational axis is equipped with a rotary encoder and the direction of the indenter can be measured with the resolution of 0.0044 degrees (16 seconds). Faces of the indenter are observed with an optical microscope and a CCD camera. Between the objective lens and the photo eyepiece, a beam splitter is placed to induce the proving beam, which is generated by the light source (semiconductor laser). The collimated light is induced through a pinhole to reduce its beam size. Then a small spot of the beam is projected on the surface of the indenter and it can be also observed with the CCD (see Fig. 6). This spot shows the position where the direction of this face is measured. If the smaller spot is used for the measurement, the measurement position can be specified more precisely and closer to the indenter tip. When the spot is located in the position distant s from the tip as shown in Fig. 5, this position of the measurement is relating to the diagonal length d and indentation depth h as follows,

$$d = 2\sqrt{2} \cdot s \cdot \sin 68^\circ \cong 2.62 \cdot s \quad (3)$$

$$h = s \cdot \cos 68^\circ \cong 0.375 \cdot s \quad (4)$$

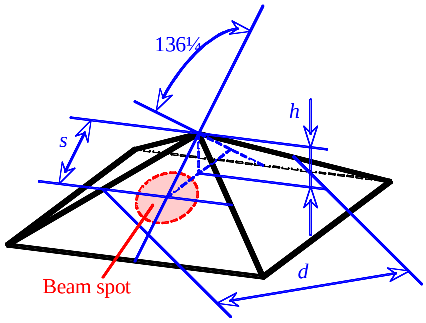


Fig. 5. Diagonal length *d*, indentation depth *h* and the length from the indenter tip along the face *s* of Vickers pyramid.

Since the diagonal length for low-force and microhardness is ranging from 20 to 166.8 μm (See Table 1), if the beam spot can be located at the position of s = 7.63 to 63.66 μm, the indenter geometry can be verified for the range of those testing force.

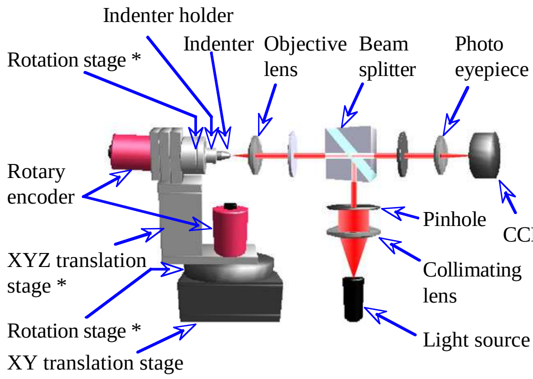


Fig. 6. Optical layout of the instrument.
* Five axes stage for indenter holder

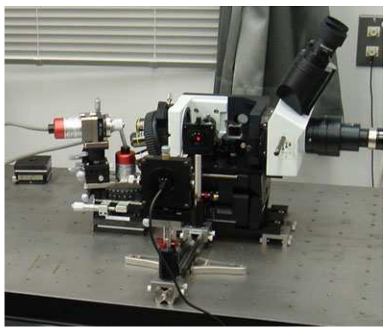


Fig. 7. Experimental apparatus.

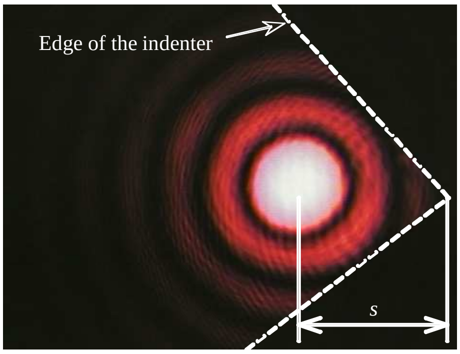


Fig. 8. Image of the light reflection on the face of Vickers pyramid.

The same Vickers indenter used for the previous experiment is measured to verify the availability of this improved instrument. The measurement results of corner angles and face angles are listed in Tables 5 to 8.

Tables 5 and 6 are the measurement results at the position of s = 150 µm where the measurement with VIVI was carried out. The results of VIVI is also listed as the reference values of angles because now VIVI is recognized as the most reliable instrument through the consideration in the previous section. These results show that the improved instrument can indicate the similar values of angles and the difference from the reference values are smaller than the expanded uncertainty of the reference values in the most cases and also it is smaller than the requirement of ISO 6507-3, *i. e.*, 0.1º for α and 0.2º for β.

Table 5. The corner angles of the square base of pyramid β measured with the improved instrument at the position of s = 150 µm.

Instrument	Objective	Angle β, degree			
		Faces 1 – 2	Faces 2 – 3	Faces 3 – 4	Faces 4 – 1
VIVI *	–	90.02	89.97	90.13	89.88
Improved instrument	5x	90.02	90.03	90.07	89.88
	10x	90.02	90.00	90.09	89.88
	20x	90.03	90.01	90.07	89.89
	50x	89.97	90.03	90.05	89.93
Maximum deviation from VIVI results		-0.05	+0.05	-0.08	+0.05

* Measurement uncertainty is 0.07º (k = 2).

Table 6. The face angles α measured with the improved instrument at the position of s = 150 µm.

Instrument	Objective	Angle α, degree	
		Faces 1 – 3	Faces 2 – 4
VIVI *	–	136.10	136.07
Improved instrument	5x	136.12	136.13
	10x	136.12	136.11
	20x	136.10	136.08
	50x	136.12	136.15
Maximum deviation from VIVI results		+0.02	+0.08

* Measurement uncertainty is 0.03º (k = 2).

Tables 7 and 8 are the measurement results at the position of s = 30 µm where the indenter is used for low-force hardness. The measured values with VIVI are also listed in the tables, however, those values are not obtained at the same location with the improved instrument. Therefore these values cannot be regarded as reference values but it would give some information to estimate the reliability of the improved instrument.

In principle, the sensitivity for detecting the normal direction of the face is getting worse with increasing the numerical aperture of the objective lens, *i. e.*, higher magnification of objective lens. However, the measured results are not so different from the results of VIVI. It seems that the improved instrument has enough performance in such a measurement range.

Table 7. The corner angles of the square base of pyramid β measured with the improved instrument at the position of s = 30 µm.

Instrument	Objective	Angle β, degree			
		Faces 1 – 2	Faces 2 – 3	Faces 3 – 4	Faces 4 – 1
VIVI *	–	90.02	89.97	90.13	89.88
Improved instrument	20x	90.00	90.02	90.05	89.93
	50x	90.01	89.98	90.09	89.92
Maximum deviation from VIVI results		-0.02	+0.05	-0.08	+0.05

* Measurement uncertainty is 0.07º (k = 2), measured at s = 150 µm.

Table 8. The face angles α measured with the improved instrument at the position of s = 30 µm.

Instrument	Objective	Angle α, degree	
		Faces 1 – 3	Faces 2 – 4
VIVI *	–	136.10	136.07
Improved instrument	20x	136.08	136.07
	50x	136.09	136.10
Maximum deviation from VIVI results		-0.02	+0.03

* Measurement uncertainty is 0.03º (k = 2), measured at s = 150 µm.

5. CONCLUSION

The geometry of Vickers indenter was measured with three instruments, which have different measurable range. It was found that the measurement uncertainty is getting greater in smaller range because of the difficulty of measurement itself and calibration of the instruments. However, each method has its own measurable range and it is useful to use several methods for understanding overall geometry of indenter in the wider range.

The improved instrument showed acceptable performance to verify the indenter geometry in the low-force hardness and it is expected that the measurement capability will be improved for this range.

REFERENCES

[1] ISO 6507-1:2005, Metallic materials — Vickers hardness test — Part 1: Test method.
[2] ISO 6507-2:2005, Metallic materials — Vickers hardness test — Part 2: Verification and calibration of testing machines.
[3] ISO 6507-3:2005, Metallic materials — Vickers hardness test — Part 3: Calibration of reference blocks.
[4] N. Hida, "Studies of Improving the Accuracy of Vickers Hardness Standard," Bulletin of NRLM Vol. 26, No. 4, pp. 175-282, 1977. (In Japanese)