

COMPARISON OF MICROSCOPIC OBSERVATION TECHNIQUES TO DETERMINE VICKERS INDENTATION DIAGONAL LENGTH

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Abstract:

In order to investigate the best way to determine the diagonal length of a Vickers indentation, bright-field (BF), dark-field (DF), differential interference contrast (DIC) and confocal scanning microscopy is utilized to observe indentations on various samples. Each of BF, DF and DIC methods has advantage and disadvantage and evaluated values should be considered carefully. 3D feature of indentation evaluated with a confocal microscope gives useful information about it. To eliminate the effect of focusing error, *focus stacking* technique is quite efficient and expected to reduce the uncertainty of measurement of diagonal length. The roughness of sample surface should be taken into account but not only the amplitude parameter, *Ra*, but also the wavelength parameter, *RSm*.

Keywords: Vickers hardness; diagonal length measurement; optical microscopy; focus stacking; confocal scanning microscopy

1. INTRODUCTION

The error of diagonal length measurement is essential factor of the uncertainty of Vickers hardness measurement. Even though, an image of indentation looks like a square marked on a flat surface, the indentation has 3D structure, and it makes perimeter of square more ambiguous.

In this study, various Vickers indentations were observed with bright field, dark field, differential interference contrast and confocal microscopy. To consider the effect of focusing error, a digital image processing technique called *focus stacking* was employed. Collected images were analyzed to discuss possible error factors of the diagonal length measurement.

2. SAMPLES, EQUIPMENT AND METHODS

Indentations were made on samples listed in Table 1 with Mitutoyo SHT-41 primary standard Vickers hardness machine and Shimadzu HMV microhardness tester. The testing force was adjusted according to sample hardness so that the size of

indentations would have similar size. The diagonal length, ranging from 77.67 μm to 102.04 μm , was measured with the measuring system on SHT-41, which were calibrated with a traceable reference standard. For the comparison of observation technique, images of each indentation were taken with Olympus BX series optical microscope with 50 \times objective lens (numerical aperture: 0.50). In order to investigate the effect of observation technique on the visibility of indentations and the measurement of the diagonal length, bright field, dark field and differential interference contrast microscopic observation were conducted for each indentation. In addition, a digital image processing technique called *focus stacking* was utilized to expand the depth of focus and improve sharpness of images. A microscopic image of an indentation was taken in every 0.5 μm in depth from the surface of sample to the bottom of indentation. Around 20 images in total, according to the indentation depth, were combined with an image processing software Adobe Photoshop to create single image in which every part is in focus. Note that this process stacks parts of images in focus in every single image only but never interfere the size of indentations (it can be done without aligning images automatically by the software). The axial position of focal plane is monitored with a 10 nm-resolution linear gauge, Mitutoyo LGH-110 Laser Hologage, attached to the microscope arm.

Table 1: List of indentations

Material	Diagonal length	Hardness		
		Average	Range	Unit
Aluminum*	101.47 μm	18.1	0.1 (0.29%)	HV0.1
Nickel*	92.62 μm	64.9	0.4 (0.64%)	HV0.3
Copper	89.86 μm	45.9	0.2 (0.37%)	HV0.2
Brass	94.85 μm	103.1	0.9 (0.89%)	HV0.5
Brass	92.46 μm	108.5	0.6 (0.54%)	HV0.5
Steel	96.63 μm	198.6	1.4 (0.71%)	HV1
Steel	77.67 μm	614.9	1.5 (0.24%)	HV2
Steel	102.04 μm	890.5	3.9 (0.44%)	HV5

* Single crystal

In addition, same indentations were observed with Lesertec HD100D confocal scanning

microscope to obtain information of three-dimensional feature of indentations. Along with 50× objective lens, 100× objective lens was used to obtain higher resolution images. Three-dimensional data of indentations were analyzed with *Wolfram Mathematica* and a 3D surface texture analysis & evaluation software *MitakaMap* of Mitaka Koki.

Table 2: Geometry of a Vickers indenter

Parameter	Value
Angle between opposite faces	136°
Angle between opposite edges	148.112°

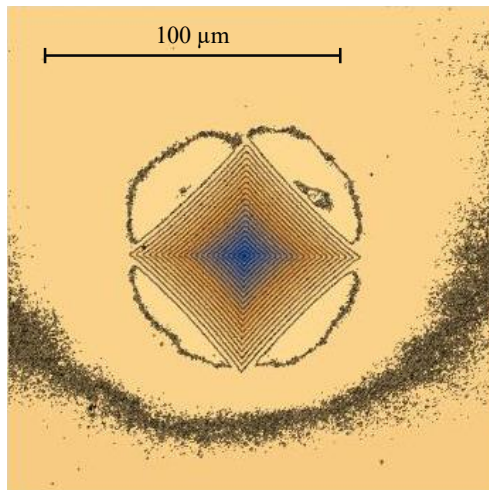


Figure 1: Typical surface deformation of indentation (steel – 614.9 HV2)

3. OBSERVATION OF INDENTATIONS

3.1. General feature of geometry of indentations

The definition of a Vickers diamond indenter is shown in Table 2. We assume that the geometry of indentation is exactly same with the indenter geometry when an indenter is in contact with sample surface under the testing force. During the plastic deformation, the volume of sample must be conserved except very small amount of change due to the elastic compression. The sample volume removed from the indentation makes *pile-up* on the surface around the indentation. After removal of the testing force, the indentation gets shallower due to elastic recovery. Therefore, the geometry of remaining impression is not exactly same with the indenter geometry. Figure 1 shows a contour plot of a typical geometry of a Vickers indentation observed with a confocal microscope. This plot shows the sample surface is piled up around the indentation. The piling up is significant in the area along with edges of the square, but the area nearby vertices is also piled up. In addition, edges are not always straight but can be concave or convex according to sample materials. The detail of analysis on confocal microscope images are discussed later. In order to investigate the detail of the geometry of

indentations, various techniques of microscopy were applied.

3.2. Bright field microscopy

The bright-field (BF) microscopy is the most common way to observe objects with an optical microscope. In a reflected light system, the light is induced through an objective lens and the reflection from the sample surface is making a magnified image on a human retina or an image sensor on a camera. The most of commercial hardness machines are equipped with this type of microscope on length measuring system. The perimeter of indentation is seen as the difference of image contrast. It is known the contrast varies with the numerical aperture (NA) of objective lens and the higher contrast is obtained with the smaller NA. Even though the image contrast is low, a larger NA, normally a higher magnification, objective lens enables measurement with lower uncertainty, according to our previous work [1].

In Figure 2, some images of indentation taken by BF microscopy are shown (photos shown without a scale bar have a field of view of $248.8 \mu\text{m} \times 186.6 \mu\text{m}$). As shown in figures, indentation perimeters do not always have a perfect square shape. In some cases, the perimeter has a barreled shape (as shown in (a)) and in other cases, it has pincushion shape (as shown in (b)). When a material has ordered structure like a single crystal, the indentation shape will be distorted circular-symmetrically (as shown in (d)). Even if the material is polycrystal, the edge of indentation can be distorted according to the grain size (as shown in (c)).

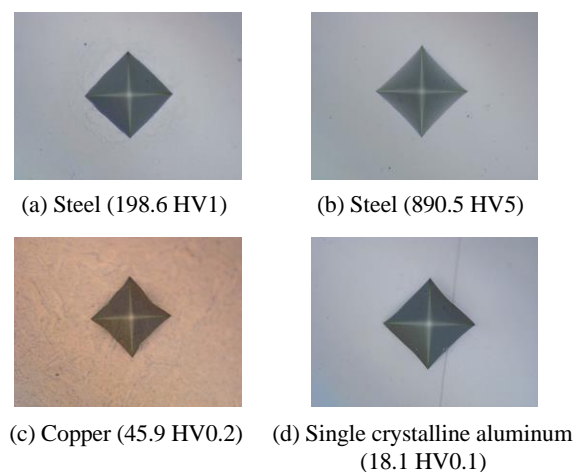


Figure 2: Bright field microscopy of indentations

3.3. Dark-field microscopy

The dark-field (DF) microscopy is the technique which collects only scattered light from the sample surface by blocking direct reflection by a light stop set in the condenser lens. The image contrast is inverted from BF, *i. e.*, the original sample surface

looks dark while an indentation looks bright. The image resolution is theoretically equal to BF.

In Figure 3, images of indentation taken by DF microscopy are shown. In the series of observations, it was found that DF microscopy is not suitable for some kinds of indentation. For an indentation with flat sample surface, a clear image could be obtained (see Figure 3 (a)), but when prominent pile-up or sink-in is occurred on sample surface, the perimeter of indentation could not be identified clearly (see Figure 3 (b)). It seemed that the DF microscopy is too much sensitive to the texture of sample surface.

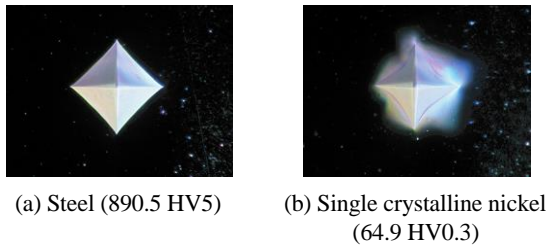


Figure 3: Dark field microscopy of indentations

3.4. Differential interference contrast microscopy

The differential interference contrast (DIC) microscopy in metallurgical microscopes is the technique which enhance the contrast due to the difference of sample height by the principle of interferometry. The image has colors according to the slope of object surface and more information can be obtained from single image. It is useful to understand what kind of surface feature exist on the surface, but a special attention must be paid for the diagonal length measurement. The contrast depends on the direction of the prism and the visibility of two diagonal could be different. So, the results may be different unless the sample is rotated before the measurement of another diagonal.

In Figure 4, examples of DIC microscopy are shown. This technique helps to observe piling up of the surface as well as the surface roughness when the sample preparation is not perfect.

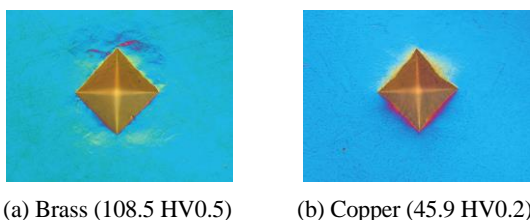


Figure 4: Differential interference contrast microscopy of indentations

3.5. Comparison between BF, DF and DIC techniques

The difference of visibility of indentation vertex is demonstrated in Figure 5. The polished surface of steel was scratched in two orthogonal directions

with 3M™ 262X Lapping Film, which has 1 μm -grit size of abrasive. Thin lines introduced on the surface in this process can be used as a guide to locate positions of a vertex. Figure 5 shows the difference of visibility of vertex from the same indentation (606.78 HV2). The position of the vertex in BF image looks slightly different from other images while the position of the vertex is almost same in DF and DIC images. The difference is approximately 0.8 μm , which is larger than the resolving power of this optical microscope, approximately 0.67 μm (wavelength of visible light 550 nm is considered). The reason is not clear, but it suggests that shallow-sloped transient area could exist around the perimeter of indentation since DF and DIC are more sensitive to such kinds of surface features.

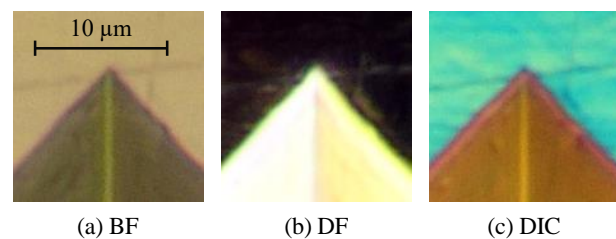
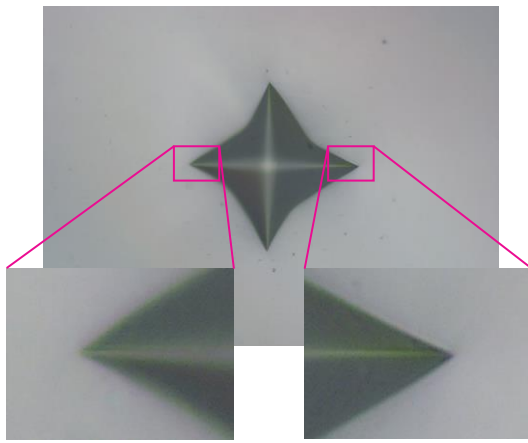


Figure 5: Difference of visibility of an indentation vertex with BF, DF and DIC techniques (steel – 606.8 HV2)

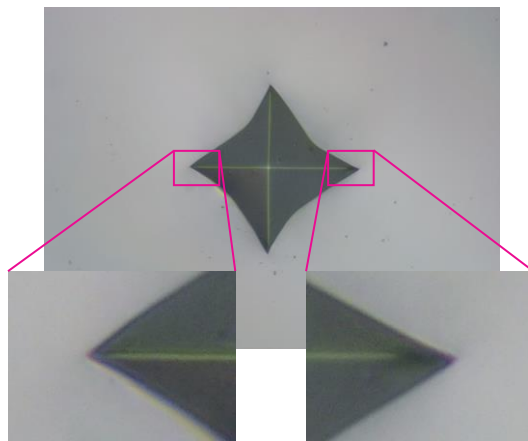
3.6. Focus stacking

The focus stacking is a digital image processing technique. When multiple images taken at the different distance from the object, *i. e.*, with focal planes at different positions, it can generate an image in which every part is in focus by combining original images [2]. Recently, this function has been equipped in commercial graphic editing software.

A BF image of an indentation on a single crystalline nickel is shown in Figure 6 (a). This sample was not perfectly aligned perpendicular to the microscope axis and has slope of approximately 0.5° along with a diagonal. This angle is much more than parallelism of 0.010 mm in 50 mm, corresponding to 0.01° , required for hardness reference blocks [3]. The slope makes a difference in focal planes at both ends of diagonal and those two points were not possible to be in focus at the same time. For this indentation, the cosine error of diagonal length is 0.004 μm which is negligible in the measurement, but the difference of focal distances is approximately 0.9 μm and it makes image blurred and will increase the uncertainty of measurement. With a focus-stacked image shown in Figure 6 (b), both ends of diagonal are in focus and it makes possible more accurate measurement.



(a) Image taken at single focal distance



(b) Focus stacked image

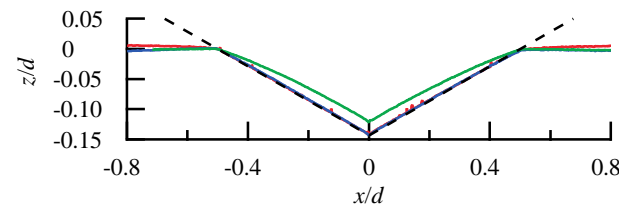
Figure 6: Image sharpening by focus stacking technique (Single crystalline nickel – 64.9 HV0.3)

3.7. Confocal scanning microscopy

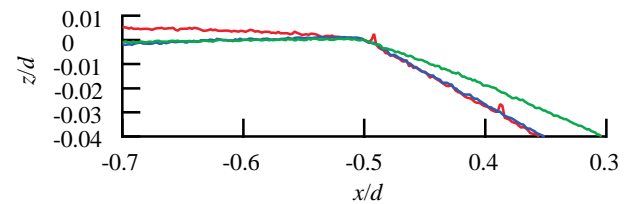
The confocal scanning microscopy has an optical layout which collect only light from the object in focus by a detector. While a sample stage moves in the direction of the axis of microscope lens, multiple confocal images of the object are obtained. By combining those images and displacement data, Three-dimensional image of the object is obtained. It should be mentioned, however, that the lateral resolution of this microscope is $0.18 \mu\text{m}/\text{pixel}$, with 50x objective, and does not have enough resolution to verify observations with other microscope techniques. On the other hand, larger geometrical feature can be evaluated quantitatively.

In Figure 7, profiles of indentations along with their diagonals are illustrated for selected copper, brass and steel samples. The scales are normalized with diagonal length so that profiles of indentations with different sizes are directly compared. The depth of indentations is shallower than expected from indenter penetration depth due to the elastic recovery. It is significant especially for 890.5 HV5 steel sample. The angle between two edges varies from the bottom of indentation to the sample surface. At the bottom of indentation, the angle is

close to the angle of indenter, and it gets wider at the surface of sample. The sample surface outside the indentation is also deformed. To compensate volume loss by the indentation, the sample surface piles up. For brass and steel samples, the highest points are at the vertices of indentation. On the other hand, the highest points are not in the field of view of the microscope for the copper sample (45.9 HV0.2). The slope of the profile makes contrast in BF microscopy. When the angle between indentation and the sample surface is steeper, obtained microscopic image will have sharp contrast, therefore, the more accurate measurement is expected.



(a) Indentation profile along g with the diagonal



(b) Magnified view around the indentation vertex

--- Indenter profile — 198.6 HV1
 — 45.9 HV0.2 — 890.5 HV5

Figure 7: Indentation profiles normalized by the diagonal length on different samples

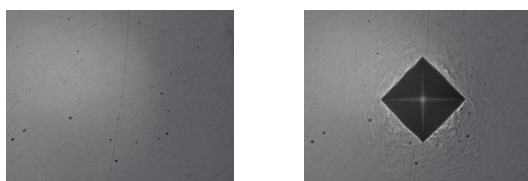
Table 3: Angle between the sample surface and the indentation edge

Material	Hardness	Angle
Aluminum*	18.1 HV0.1	163.016°
Nickel*	64.9 HV0.3	165.016°
Copper	45.9 HV0.2	166.046°
Brass	103.1 HV0.5	163.178°
Brass	108.5 HV0.5	164.083°
Steel	198.6 HV1	164.685°
Steel	614.9 HV2	164.804°
Steel	890.5 HV5	166.111°
Indenter profile		164.056°

* Single crystal

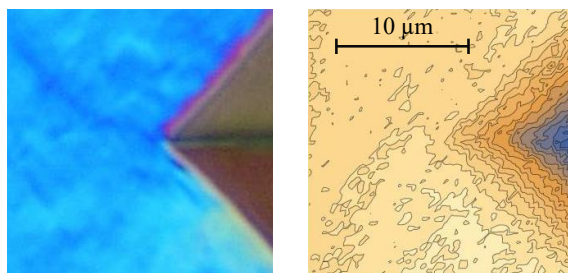
The ISO standard [3] says that the surface of a reference block must be smooth and the roughness shall be better than $Ra = 0.05 \mu\text{m}$. Even though the sample was prepared conformingly to this requirement, the surface get roughened after indentation on some samples (see Figure 8) and it makes more difficult to identify the position of indentation vertex. This phenomenon was observed on softer samples but not on single crystals. It

suggests samples with coarse grain material might behave this deformation. A DIC image and corresponding contour plot from confocal microscopy are shown in Figure 9. The analysis on the contour plot with *MitakaMap* software shows that the roughened surface close to the indentation vertex on the steel sample has surface texture of $Ra = 0.024 \mu\text{m}$ and $RSm = 1.679 \mu\text{m}$, where Ra is the arithmetic mean deviation of the assessed profile and RSm is the mean width of the profile element [4]. They are amplitude and wavelength in other words. Even though the Ra number is small enough, the visibility of indentation is affected by the surface roughness and it might be better if another parameter like RSm is considered for the evaluation of measurement uncertainty of diagonal length.



(a) Original surface (b) Surface after indentation

Figure 8: An example of the surface texture change on a steel sample (at 200 HV1 hardness level)



(a) DIC image (b) Contour plot from confocal microscopy

Figure 9: Indentation-induced surface roughening (steel – 198.6 HV1)

4. CONCLUTIONS

In this paper, different methods of microscopic observation, BF, DF and DIC are compared along with the confocal scanning microscopy. Since the indentation has complex 3D feature, understandings of characteristics of methods are necessary for appropriate diagonal length measurement. From the comparison between BF, DF and DIC technique, it was suggested that the position of indentation vertex in BF is slightly different from other technique and the diagonal length evaluated from BF technique might be smaller. The focus of an image is the key to determine both ends of the diagonal. It was demonstrated that the focus stacking technique is quite useful to improve the visibility of the indentation vertices. The surface roughness, another significant uncertainty sources, should be considered with not only amplitude parameter, Ra , but also wavelength parameter, RSm .

5. REFERENCES

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