Correlation between Nanoindentation Test Result and Vickers Hardness

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Abstract − The nanoindentation method is one of the recently developed hardness testing methods, and was standardized as ISO14577 in 2002. Recently, papers and reports using the analysis method and parameters defined in ISO14577 have been published. Among those parameters, indentation hardness is used pretty frequently. This is probably because indentation hardness is deemed to be correlated with Vickers hardness, and sometimes indentation hardness is treated as Vickers hardness. Certainly, in the papers cited in ISO14577, it can be confirmed that indentation hardness almost coincides with Vickers hardness. However, in those cited papers, only some general metals and fused silica were used as specimens of nanoindentation. Actually, in addition to general metals and fused silica, various materials such as resin materials and amorphous materials should become specimens of nanoindentation. Accordingly, it is described in this paper whether or not the correlation between nanoindentation hardness and Vickers hardness can be applied to such various materials, and an analysis method that has stronger correlation with Vickers hardness is also described.

Keywords: Indentation hardness, Vickers hardness, Indentation work

1. INTRODUCTION

Nanoindentation method is remarked as one of the evaluation methods of mechanical properties of thin-film materials that are important elements supporting the advanced technologies [1], and demands for the nanoindentation method will further increase. Under the circumstances, researches for standardizing the nanoindentation method have been also performed, and international standard ISO14577 about instrumented indentation test was established in 2002 [2]. However, although in this international standard, Martens hardness "HM", indentation hardness "H_{IT}", indentation modulus "E_{IT}", indentation creep "C_{IT}", indentation relaxation "R_{IT}", and indentation work ratio "η_{IT}" are defined as materials parameters that are analyzed from the relationship curve between test force "F" and indentation depth "h", these materials parameters are not recognized enough in industrial fields, and reviews of analysis method and researches for new analysis methods are now being performed. On the other hand, recently, there are papers that discuss examples of evaluating new materials by using these parameters. This means that the nanoindentation method is utilized not only in the hardness research field, but also purely in the material development field where "hardness" is required as a value indicating material properties.

In addition, among the before-mentioned parameters, it is increased that indentation hardness is used to evaluate thin-film materials, for which Vickers hardness has not been able to be represented. This suggests that also in thin-film materials, resistance to plastic deformation represented by Vickers hardness is an important property. However, indentation hardness is often treated as a value equivalent to Vickers hardness instead of discussing the correlation between them, and the description "Vickers hardness obtained by nanoindentation" often appears in papers and reports. The analysis method of indentation hardness defined by ISO14577 was proposed mainly by Oliver and Pharr [3], and according to their research, it was confirmed that indentation hardness preferably coincides with Vickers hardness. However, as specimen in their research, only several kinds of general metals and fused silica, which represent clear elasto-plastic behavior, were used. On the other hand, target materials of nanoindentation should include various other materials such as rubbers, resins and amorphous materials [4], and it is not clear whether or not their analysis method can be applied to those other materials. Accordingly, the present experiments have been performed to examine whether or not the correlation between indentation hardness and Vickers hardness can be also applied to rubbers, resins and amorphous carbon (DLC).

2. EXPERIMENTS

2.1. Testing Machine

For nanoindentation test, Mitutoyo-made micro zone testing system "MZT-500" is used. Figure 1 shows the appearance of "MZT-500 Series". "MZT-500" consists of a main unit, a controller, and a personal computer. Furthermore, "MZT-500" is normally provided with a vibration isolating mechanism for eliminating influences by external vibrations, and also normally provided with a cabin cover for eliminating influences by external winds and noises. These measures are indispensable for realizing micro-zone hardness test. A balance lever mechanism that is robust against disturbance vibrations is adopted for the loading mechanism, and the test force is generated by a

combination of an electromagnetic coil and a permanent magnet. The indentation depth is measured by a capacitance type displacement sensor arranged in the upper side of the indenter and aligned with the indenter.

The basic operations are performed by the personal computer, and the personal computer is loaded with software for calculating materials parameters defined in ISO14577.

Fig. 1. Mitutoyo-made micro zone testing system "MZT-500 Series"

2.2. Specimens and Testing Conditions

Table 1 shows the list of tested specimens. Vickers hardness is measured by Mitutoyo-made micro hardness testing machine "HM-221". All the specimens are tested by "MZT-500" with a test force of 10 mN. The loading time, the holding time and the unloading time of the test force are 10 s – 10 s – 10 s, and the test is performed at three points for each specimen.

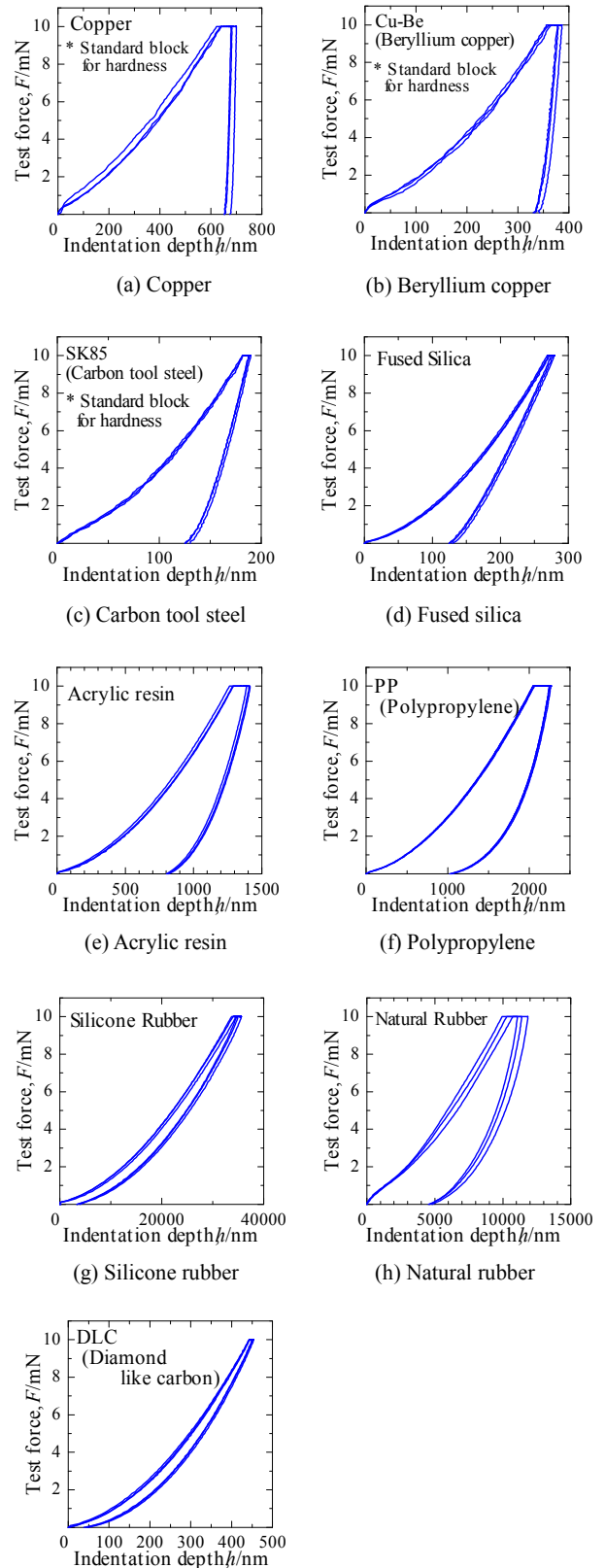Table 1. List of tested specimens

	Vickers hardness, HV [N/mm^2]	Remarks
Copper	412	Standard block for hardness
Beryllium copper (Cu-Be)	1960	Standard block for hardness
Carbon tool steel (SK85)	8790	Standard block for hardness
Fused silica	8040	Synthetic silica
Acrylic resin	206	Thickness of plate: 3 mm
Polypropylene (PP)	74	Thickness of film: 0.25 mm
Silicone rubber	20	Thickness: 1 mm
Natural rubber	10	Thickness: 2 mm
DLC (Diamond-like Carbon) film	30000	Thickness > 10 μm

3. RESULTS AND DISCUSSION

3.1. Test Result for Each Specimen

In each of Fig. 2 (a) to (i), test results performed three times for each specimen are shown in one graph. Concerning each specimen, three measurement results coincide with each other very well. DLC represents elastic

behavior where the loading curve almost coincides with the unloading curve.

(a) Copper

(b) Beryllium copper

(c) Carbon tool steel

(d) Fused silica

(e) Acrylic resin

(f) Polypropylene

(g) Silicone rubber

(h) Natural rubber

(i) DLC (Diamond-like Carbon) film

Fig. 2. Test result for each specimen

3.2. Relationship between Indentation Hardness "H_{IT}" and "HV"

Figure 3 shows the relationship between the indentation hardness "H_{IT}", which has been obtained by analyzing the test result shown in Fig.2, and "HV". The straight line shown in Fig. 3 represents an assumption that "H_{IT}" and "HV" have relationship described below using a coefficient C_1.

$$H_{IT} = C_1 HV \qquad (1)$$

The equation (1) can apply to the tested specimens other than silicone rubber, natural rubber and DLC by using 1.25 as C_1. The following items are considered as reasons why C_1 is not 1.0, namely why "H_{IT}" is not equal to "HV".

(a) To calculate "H_{IT}", not the surface area of the indentation, but the projected area is used.
(b) Error in detecting the zero point (namely, the instant when the indenter starts to contact the specimen surface)
(c) Micro truncation or rounded shape of the indenter tip
(d) The specimen surface is actually harder (Work hardening).
(e) Size effect about hardness (As the indentation depth is smaller, the hardness increases.)
(f) In principle, "H_{IT}" does not coincide with "HV".

Concerning the above (a), when the projected area is converted into the surface area based on the geometric form of the indenter, C_1 becomes 1.13, but C_1 does not become 1.0. Concerning the above (c), a method, where correction is performed in such a manner that the elastic modulus of the specimen obtained by calculation keeps a constant value, has been proposed. However, since this correction method has not been established as a reliable method, and since in the present research, error in hardness caused by error in the indenter tip shape in an order of nanometer is not discussed, "H_{IT}" is calculated without performing the correction. Generally speaking, concerning the tested specimens other than DLC, silicone rubber and natural rubber, there is correlation between "H_{IT}" and "HV" although "H_{IT}" does not coincide with "HV".

In Fig. 3, concerning polypropylene, silicone rubber and natural rubber, since indentation formed by Vickers hardness test is difficult to observe accurately, the reading error of the indentation length is probably large, and accordingly an error bar containing an error of 50% is attached to measurement value. Furthermore, concerning DLC, since indentation is very small, the reliability of Vickers hardness value is probably low, and accordingly an error bar corresponding to the range described in the reference [5] is attached. However, even while considering such error bars, silicone rubber, natural rubber and DLC are not represented by the above equation (1). This means that concerning these materials, indentation hardness defined by ISO14577 does not correlate with Vickers hardness. Namely, for some materials, it is not reasonable to consider that "H_{IT}" is equivalent to "HV".
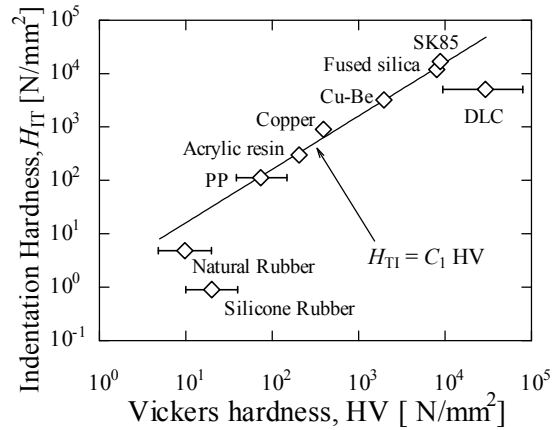

Fig. 3. Relationship between "H_{IT}" and "HV"

3.3. Discussion based on Plastic Deformation Work "W_{Plast}"

ISO14577 defines indentation work ratio "η_{IT}". The indentation work ratio is obtained as a ratio of the indentation work by elastic deformation "W_{elast}" to the mechanical work caused during the indentation process "W_{total}". "W_{total}" is summation of "W_{elast}" and the indentation work by plastic deformation "W_{plast}", and each work is represented by the area of the corresponding part shown in Fig. 4.
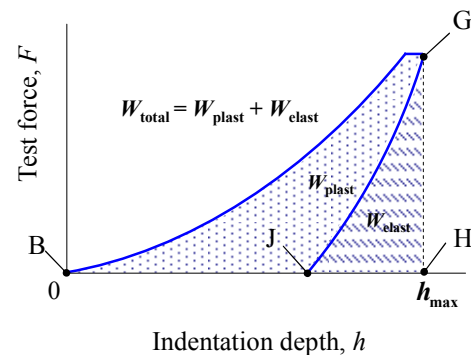

Fig. 4. Works obtained by test result

W_{total}: Area of the part surrounded by a curve extending through the points B, G and H.
W_{plast}: Area of the part surrounded by a curve extending through the points B, G and J.
W_{elast}: Area of the part surrounded by a curve extending through the points J, G and H.

Namely, "η_{IT}" is represented by the following equation.

$$\eta_{IT} = \frac{W_{elast}}{W_{total}} \times 100 \qquad (2)$$

As shown in the following equation, each work "W" (area of each part) is obtained by summing the product of

the test force and the indentation depth in each micro interval of indentation depth.

$$W = \int Fdh \qquad (3)$$

In the above equation, when the test force is same, the indentation work by plastic deformation "W_{plast}" is proportional to the plastic deformation depth. And the indentation area by Vickers hardness test "A" is proportional to the square of the plastic deformation depth. Accordingly, "W_{plast}" and the indentation area "A" have a relationship represented by the following equation while a coefficient C_2' is used.

$$W_{plast} = C_2' \sqrt{A} \qquad (4)$$

Furthermore, Vickers hardness is obtained by dividing the test force by the indentation area. Accordingly, by using a coefficient C_2 and Vickers hardness "HV" in the above equation (4), the following equation is obtained.

$$W_{plast} = C_2 \frac{1}{\sqrt{HV}} \qquad (5)$$

Figure 5 shows the result of the relationship between "W_{plast}" and "HV". The straight line shown in Fig. 5 represents the above equation (5). As shown in Fig. 5, for all the tested specimens, there is high correlation between the indentation work by plastic deformation "W_{plast}" and Vickers hardness "HV". Accordingly, when evaluating "HV", it is effective to use "W_{plast}".
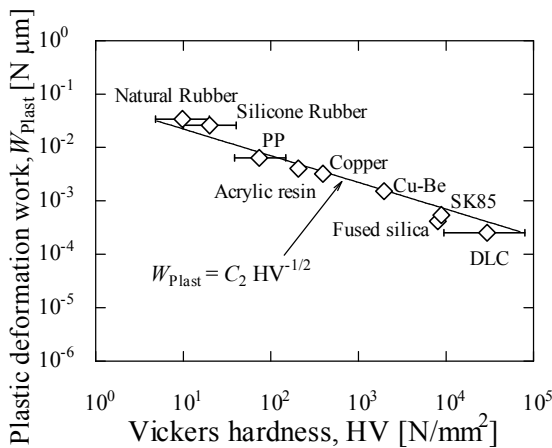

Fig. 5. Relationship between "W_{plast}" and "HV"

4. CONCLUSION

From the facts described above, the following conclusions are obtained.

- Concerning fused silica, carbon tool steel, beryllium copper, copper, acrylic resin and polypropylene, the indentation hardness is correlated with Vickers hardness.

- Concerning silicone rubber, natural rubber and DLC, the indentation hardness is not correlated with Vickers hardness.
- Concerning all the tested specimens (fused silica, carbon tool steel, beryllium copper, copper, acrylic resin, polypropylene, silicone rubber, natural rubber and DLC), the indentation work by plastic deformation "W_{plast}" is correlated with Vickers hardness.
- When evaluating Vickers hardness "HV", it is effective to use the indentation work by plastic deformation "W_{plast}".

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