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DIFFERENCES OF PERFORMANCE BETWEEN THE STEEL STANDARD BLOCKS FOR HARDNESS MADE IN JAPAN AND THAT IN GERMANY IN THE DISPLACEMENT RANGE FROM NANO TO MICROMETER

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Abstract – Even though the efforts have been made to develop an ideal hardness testing machine, it is inevitable that the difference of performance between the models (or types) of testing machine significantly affect the test results especially in the range of nanoindentation. The hardness reference blocks can be used to compensate the unreliability of machines by evaluating the difference between machines. The performance of hardness reference blocks should be considered from various aspects, *e. g.*, repeatability and uniformity of parameters such as *HM* or H_{IT}, easy handling, durability of testing, the dimensions and the price. Due to the large amount of usage in the world industry, the hardness reference blocks of Japanese manufacturer (HMV900, HMV500, UMV500, UMV700 of YSTL) and German manufacturer (HV840 and HV540 of Buderus) are chosen in this report and tested by using PICODENTER HM500 nanoindentation machine of Helmut Fischer, which can cover the displacement range of pico- to micrometers.

Keywords: nanoindentation, hardness reference block, coefficient of variation

1. INTRODUCTION

The *hardness reference block* is the reference material that has the standard value for hardness defined by calibration laboratories. By using the blocks to verify hardness testing machines, the throughout performance of machines which is affected by many factors can be clearly evaluated on-site.

In these days, *Nanoindentation*, the hardness test applicable to the displacement range of less than 1 μm, is getting popular as a new technique of material test in cutting-edge industries like nanotechnology. Since it is necessary to correct device constants such as frame compliance, indenter geometry, etc. to obtain reliable test results with nanoindentation, reference blocks play an

Table 1 Specification of the specimens

No.	Manufacturer	Symbol	Hardness value of standard blocks for hardness	*HM*	H_{IT}	Composition	Young's Modulus	Poisson's Ratio	Size
				N/mm^2	N/mm^2		*Es* [GPa]	ν_s	[mm]
1	Helumut Fisher GmbH+Co.KG	BK7 (Glass)	-	4124.6 (300mN)	8240 (300mN)	B_2O_3-SiO_2	79.2	0.214	50×50×t10.1
2	Yamamoto Scientific Tool Laboratory Co.LTD. (YSTL)	HMV500	HMV499 (98mN)	-	-	C:0.8～0.9%	210	0.3	ϕ25×t5
3		HMV900	HMV905 (98mN)	-	-				
4		UMV500	UMV500 (0.01kgf) (98mN) UMV508 (0.001kgf) (9.8mN)	4712 (9.8mN)	6113 (9.8mN)				ϕ25×t6
5		UMV700	UMV702 (0.01kgf) (98mN) UMV712 (0.002kgf) (19.6mN)	6415 (9.8mN)	9086 (9.8mN)				
6	Edelstahlwerke Buderus AG	HV540	-	5044 (1000mN)	6887.3 (1000mN)	C:0.75% Si:0.299% Ma:1.941%			△35×t6.1 regular triangle
7	Edelstahlwerke Buderus AG	HV840	-	6701.4 (1000mN)	10236.3 (1000mN)				

important role as reference standards.

The most important property of hardness reference block is uniformity of hardness. Due to very small depth and size of indentations, measurement values are affected by metallurgical microstructure or crystal orientation. In order to avoid these influences, glass materials such as fused silica or BK7 have been preferably used as reference materials because those materials do not have any structure inside. However, in order to verify testing machines in wide range of hardness corresponding to the target materials in nanoindentation test, other appropriate materials are expected as reference materials.

Steel reference blocks, generally used in micro range of hardness test, cover hardness range from raw steel material to hardened high carbon steel. The attempt to refine the grain size of steel blocks has already made to realize the uniform hardness without showing the size effect even for small indentations and this technique is applied to the fabrication of blocks for microhardness. Recently, steel blocks which adapt to smaller indentations have been developed and applied to nanoindentation.

In this report, steel-made hardness reference blocks of Japanese and German manufacturers are examined in accordance with the procedure of ISO14577-1 and their characteristics are compared. In the experiment, two types of blocks, HMVs and UMVs, of Yamamoto Scientific Tool Laboratory Co. LTD., Japan and Micro-Blocks of Edelstahlwerke Buderus AG, Germany are measured with a nanohardness testing machine, PICODENTOR HM500 of Helmut Fischer GmbH + Co.KG, Germany and the results are analyzed.

2. EXPERIMENTS

2.1. Specimens

The reference blocks used for the experiments from nano- to micrometer displacement range are listed in Table 1.

The block No.1 (BK7) is the reference block provided by the machine manufacturer as the reference standard for the measurement system. The blocks No.2 (HMV500), No3 (HMV900), No.4 (UMV500) and No.5 (UMV700) are the steel reference blocks manufactured by Yamamoto Scientific Tool Laboratory (YSTL), Japan. The blocks No.6 (HV540) and No.7 (HV840) are the steel reference blocks manufactured by Edelstahlwerke Buderus, Germany.

Both series of YSTL blocks, *i. e.*, HMV and UMV, are made of high-carbon tool steel SK85 (JIS). The former is the reference block for the microhardness and calibrated by the manufacturer with HV 1, HV 0.1 and HV 0.01 scales. The latter is prepared for the microhardness with much lower testing force or the instrumented indentation test. UMV 500 is calibrated by the manufacturer with HV 0.01 and HV 0.002 scales while UMV 700 is calibrated with HV 0.01 and HV 0.001 scales. In addition, the manufacturer provides the test results of *HM* and the indentation curve at 9.8 mN of the maximum testing force in accordance with ISO 14577-1.

The surface areas and the prices of the reference blocks are listed in Table 2 (There is not a distributor of Buderus in Japan. The price of Buderus' block shown in this table is estimated possible price in Japan).

Table 2 Surface area and price of each specimens

	HMV	UMV	HV
Manufacture	YSTL		Buderus
Surface area (mm^2)	490.9		530.4
Price (Yen)	¥50,000	¥100,000	¥100,000

2.2. Indenter

The practical model of Vickers indenter from nano to micrometer displacement range is illustrated in Fig. 1. The symbol α represents the angle between opposite faces, which is defined as 136°. Fig. 2 (a) and (b) show the cross sections of AFM images of the indenter measured by MPA/NRW and authors, respectively. α is evaluated as 135.51° with the result (a); α is 134.68° with the result (b). This result of α corresponds to 22 nm of the truncation length, Δh.

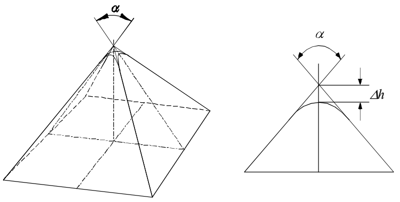
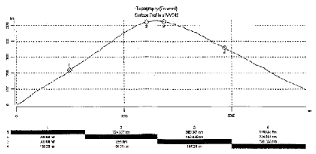
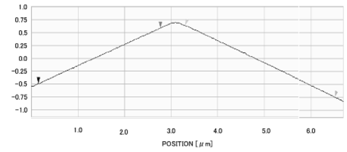


Fig. 1 Practical Microvickers Indenter



(a) 2-D AFM profile offered by MPA/NRW



(b) 2-D AFM profile measured by authors

Fig. 2 AFM profile of Microvickers indenter tip

2.3. Testing machine

PICODENTOR HM500, manufactured by Helmut Fischer GmbH + Co. KG, Germany, is used for the experiments [Fig. 3]. The specifications of the machine are as follows:

Testing force range: 0.005 to 500 mN
Testing force resolution: ≤ 100 nN
Indention depth resolution: ≤ 40 pm
Accuracy of test location: ± 3 μm
The maximum sample size: 190 × 133 × 133 mm
The maximum weight of sample: 9.8 N
Overall magnification of optical device: 40×, 200× and 400×
The maximum indentation depth: 150 μm

The instrument is installed on the active vibration isolation table TS-140 manufactured by the Table Stable, Ltd., Switzerland.

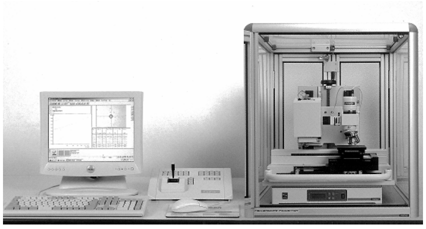


Fig. 3 Testing machine Fischer PICODENTER HM500

2.4. Experimental condition

The experiments were carried out with seven reference blocks listed in Table 1 under 12 different testing forces of 0.098 mN, 0.245 mN, 0.49 mN, 0.98 mN, 2.45 mN, 4.9 mN, 9.8 mN, 24.5 mN, 49 mN, 98 mN, 245 mN and 490 mN.

3. EQUATIONS FOR *HM*, H_{IT}, E_{IT}

3.1. Equation of Martens hardness (HM)

Martens hardness HM is defined as the testing force F divided by the contact surface area $A_s(h)$ of the indenter penetrating the depth of h,

$$HM = \frac{F}{A_S(h)} = \frac{F}{26.43 \times h^2}. \tag{1}$$

For Vickers indenters, the surface area can be calculated with the following equation (area function)

$$A_S(h) = \frac{4\sin(\alpha/2)}{\cos^2(\alpha/2)} \cdot h^2, \tag{2}$$

where α is the angle between opposite faces of Vickers pyramid and ideally to be 136°.

3.2. Equation of indentation hardness (H_{IT})

Indentation hardness H_{IT} is a measure of the resistance to permanent deformation. It is calculated from the maximum testing force F_{max} divided by the projected area of contact A_p between the indenter and the test piece as the following equation,

$$H_{IT} = \frac{F_{\max}}{A_p}. \tag{3}$$

A_p is determined from the force-displacement curve by considering the elastic recovery of test surface.

$$A_P = 4\tan^2\frac{\alpha}{2} \cdot h_C^2 = 24.50 \cdot h_C^2 \tag{4}$$

The parameter h_c is the estimated depth of contact under the testing force F_{max} and calculated with the following equation,

$$h_c = h_{\max} - \varepsilon(h_{\max} - h_r). \tag{5}$$

where h_{max} is the maximum indentation depth at F_{max} and h_r is the intersection point of the tangent to the unloading curve and the indentation depth-axis. ε is the correction factor and to be 3/4 for Vickers indenter (See Fig. 4).

3.3. Equation of indentation modulus (E_{IT})

Indentation modulus E_{IT} is calculated from the slope of the tangent to the unloading curve as shown in Fig. 4 and other parameters such as A_p according to following equations,

$$E_{IT} = \frac{1 - \nu_s^2}{\frac{1}{E_r} - \frac{1 - \nu_i^2}{E_i}} \tag{6}$$

$$E_r = \frac{\sqrt{\pi}}{2C\sqrt{A_p}} \tag{7}$$

where

ν_s is the poisson's ratio of the test piece

ν_I is the poisson's ratio of the indenter

E_r is the reduced modulus of the indentation contact

E_i is the modulus of the indenter

C is the compliance of the contact [$=1/S$]

S is the slope of the tangent of the force vs. indentation depth curve

A_p is the projected contact area, value of the indenter area function at the contact depth defined in Section 4.6 of ISO14577-2:2002

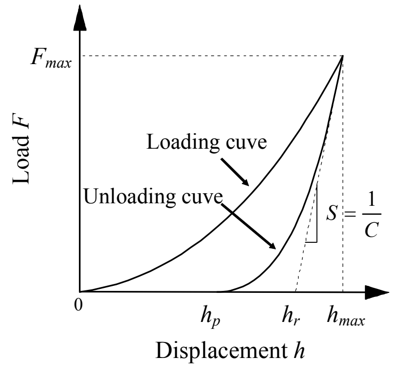
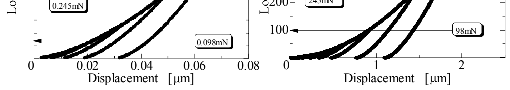


Fig. 4 Schematic illustration of indentationload-displacement data

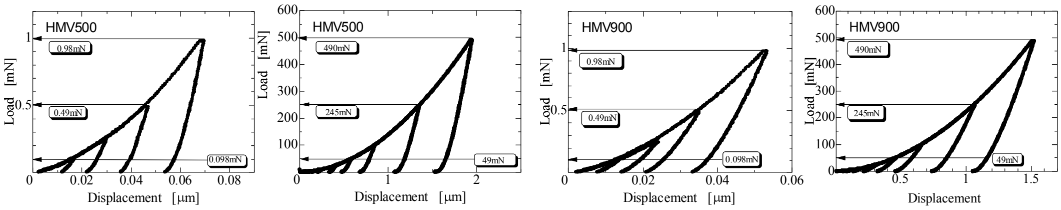
4. EXPERIMENTAL RESULTS

Fig. 5 shows the indentation curves obtained from the indentations of seven reference blocks. The parameters of *HM*, H_{IT} and E_{IT} are evaluated from the indentation curves and their average values are shown in Fig. 6.

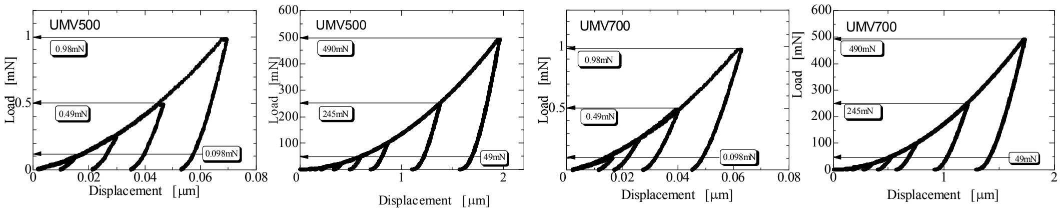


(a) Loading and unloading curves for BK7 (Fischer)

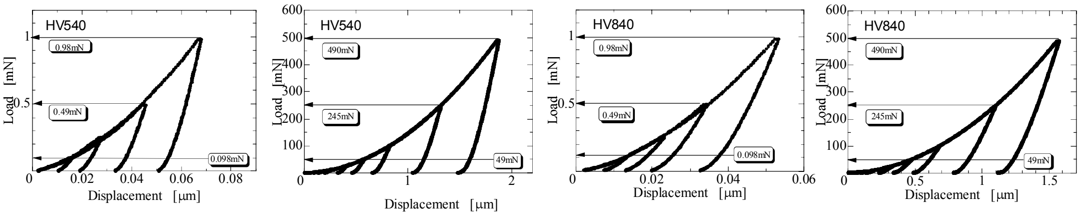
Fig. 5 Loading and unloading curves for all specimens



(b) Loading and unloading curves for HMV (YSTL)



(c) Loading and unloading curves for UMV (YSTL)



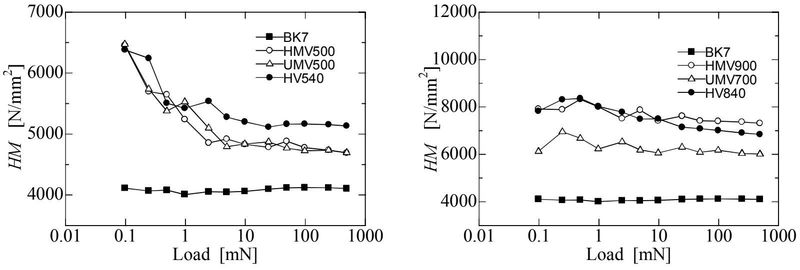
(d) Loading and unloading curves for HV (Buderus)

Fig. 5 Loading and unloading curves for all specimens

5. DISCUSSION

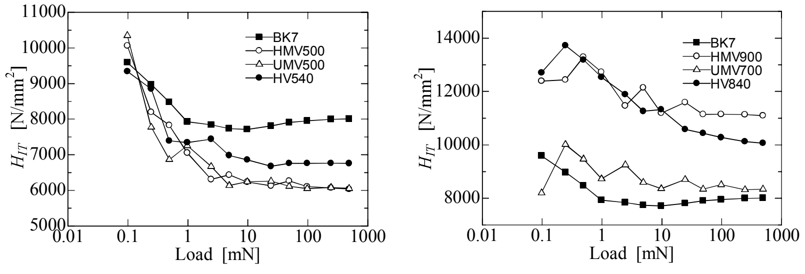
The testing machine used in this study is calibrated by using BK7 reference block. The obtained data is corrected according to the equation so that the calculated value of *HM* for BK7 is to be constant all over the testing force range. This is the reason why *HM* is constant in Fig. 6 (a) and (b). In principle, H_{IT} and $E_{\mathrm{IT}}/(1-\nu_s^2)$ should be constant for BK7. However, the results in Fig. 6 (c) to (f) show that the former is increasing for smaller testing force while the latter is decreasing.

The reference blocks of both manufacturers showed good



(a) *HM* for BK7, HMV500, UMV500. HV540

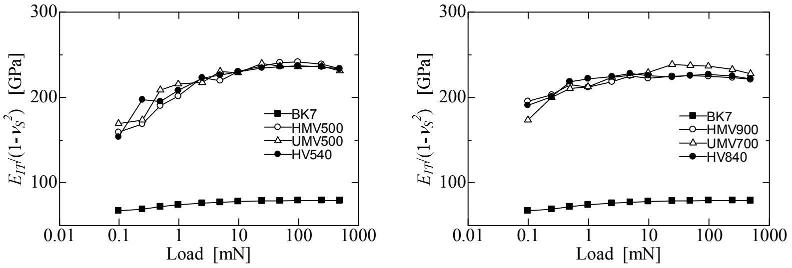
(b) *HM* for BK7, HMV900, UMV700. HV840



(c) H_{IT} for BK7, HMV500, UMV500. HV540

(d) H_{IT} for BK7, HMV900, UMV700. HV840

Fig. 6 Values of *HM*, H_{IT} *and* $E_{IT}/(1-\nu_S^2)$



(e) $E_{IT}/(1-\nu_S^2)$ for BK7, HMV500, UMV500. HV540 (f) $E_{IT}/(1-\nu_S^2)$ for BK7, HMV900, UMV700. HV840

Fig. 6 Values of *HM, H_{IT} and E_{IT}*$/(1-\nu_S^2)$

performance. To investigate the difference between those reference blocks in detail, the coefficient of variation (CV), *i.e.*, the standard deviation divided by respective average value, was calculated for each reference block at each testing condition. Calculated CVs were plotted with respect to the maximum load as power functions obtained through the least square regression (See Fig. 7) so that the general trends of those repeatability/uniformity can be clearly seen.

5.1. Comparison between Japanese blocks (YSTL: HMV, UMV) and German blocks (Buderus : HV)

5.1.1. Comparison in HM and H_{IT}

In all cases, CV is significantly increasing in the condition of smaller testing forces. As an example, if the CVs of *HM* of 500 HV level at 0.98 mN of testing force are compared, CVs are increasing in the order of 5.0 % for UMV500 (YSTL), 9.4 % for HMV500 (YSTL) and 8.0 % for HV540 (Buderus), respectively (see Fig. 7 (a)).

The CV of BK7 block is remarkably smaller than those of other blocks. Generally speaking, the contributions of the machine and the block to the measurement uncertainty of hardness test cannot be separated. The result on BK7 block, however, suggests that the variation of the testing machine itself is relatively small in respective of repeatability.

At the testing force of 490 mN (the maximum testing force in this study), CVs of all reference blocks are comparable and approximately 1 %. For the smaller testing force, the difference between reference blocks increases. It can be concluded that the YSTL blocks are more stable in all testing conditions than the blocks of another manufacturer.

5.1.2. Comparison in E_{IT} $/(1-\nu_s^2)$

The similar trends of hardness parameters can be seen for the indentation modulus, $E_{IT}/(1-\nu_s^2)$, *i. e.*, the values are significantly increasing with decreasing testing force. However, there is only one difference between the middle and the high hardness levels. At the middle hardness level, YSTL block (UMV500) shows the highest value, whereas at the high hardness level, Buderus block (HV840) is the highest.

At the middle hardness level with 0.98 mN of testing force, CV of HV540 (Buderus) is approximately 6.8 % whereas those of HMV500 (YSTL) and UMV500 (YSTL) are approximately 5.0 % and 9.3%.

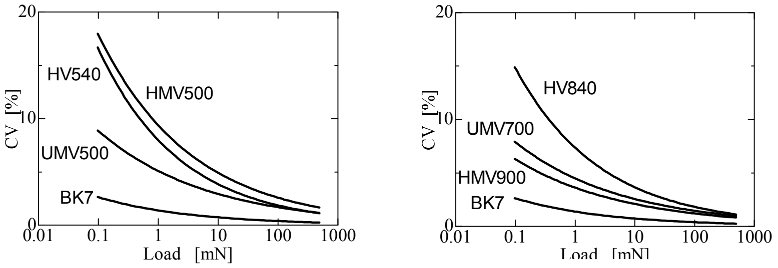
At the high hardness level the differences between blocks are less significant than at the middle hardness level.

5.2. Comparison between different hardness levels

In this report, the reference blocks are divided into two groups. One is the middle hardness level (500 to 540 HV) and another is the high hardness level (700 to 900 HV).

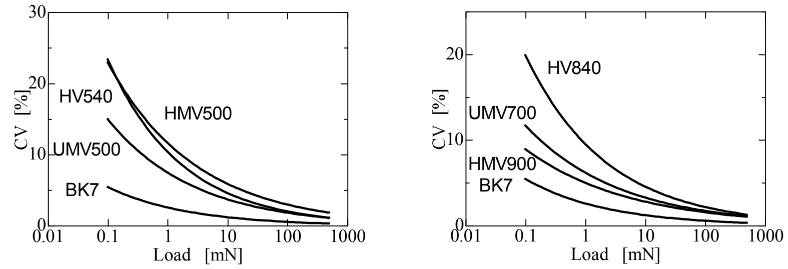
It is found that the differences of CVs between manufacturers are not so large if those are compared with the differences between different hardness levels, *e. g.*, 500 HV and 900 HV, of the same manufacturer.

The possible reason is that the reference blocks of both manufacturers are made by tempering quenched steel blocks to obtain the desirable hardness. These heat treatments affect the microstructure of reference blocks. It can be supposed that the reference blocks at the high hardness level have finer and more uniform grains of martensitic phase as results of the heat treatment.



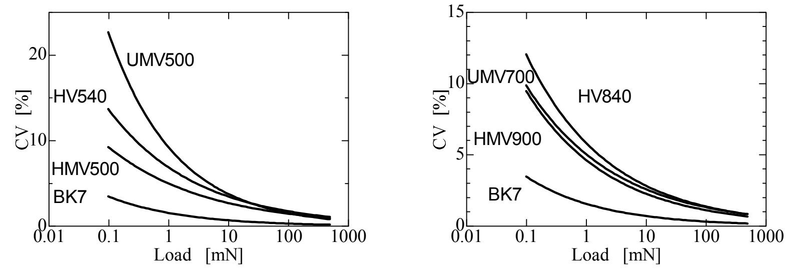
(a) *HM* for BK7, HMV500, UMV500. HV540 (b) *HM* for BK7, HMV900, UMV700. HV840

Fig. 7 Coefficient of variation curves for specimen's characteristics



(c) H_{IT} for BK7, HMV500, UMV500. HV540

(d) H_{IT} for BK7, HMV900, UMV700. HV840



(e) $E_{IT}/(1-\nu_S^2)$ for BK7, HMV500, UMV500. HV540

(f) $E_{IT}/(1-\nu_S^2)$ for BK7, HMV900, UMV700. HV840

Fig. 7 Coefficient of variation curves for specimen's characteristics

6. CONCLUSION

(1) In comparison of CVs of hardness values, *HM* and $H_{\rm IT}$, between high hardness level (UMV700 and HV840) and middle hardness level (UMV500 and HV540), CVs of hardness values, *HM* and $H_{\rm IT}$, are smaller at the high hardness level.
(2) The similar trend as mentioned above is more significant for YSTL reference blocks.
(3) The prices of YSTL's HMV and UMV blocks are 50,000 yen and 100,000 yen, respectively. The calibration certificates are attached to the both blocks. In addition, the indentation curve under the maximum testing force of 9.8 mN is attached to UMV blocks.
(4) The test results of instrumented indentation are reported in the calibration certificate attached to Buderus' blocks. The expected price of the block is 100,000 yen in Japan.
(5) The area of test surface of Buderus' blocks is 1.08 times larger than that of YSTL's blocks.
(6) As far as the experiments with the testing force range examined in this study are concerned, it can be concluded that the YSTL's blocks have the advantage of better uniformity, *i.e.*, smaller CVs, of *HM* and $H_{\rm IT}$ and lower price.

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