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DEVELOPMENT OF HIGH-ACCURACY HARDNESS STANDARD BLOCKS IN JAPAN AND FUTURE OUTLOOK

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Abstract – The sources of problems with a hardness testing machine that are detectable via day-to-day inspection using hardness standard blocks, or indirect verification, can be roughly divided into three: the indenter, the test force, and the indentation measuring apparatus. If any problems are detected, repair or recalibration of the testing machine, focusing on the aforementioned three factors, is carried out based on the results of verifying the direct accuracies of that machine. These efforts have been accumulated to acquire the high level of hardness testing technology as is available now.

This article describes the development of a hardness standard block as a tool for indirect verification of hardness testing machines, discusses possible factors that have effects on hardness measurements, and provides insights into the future hardness tests from a perspective of a hardness block manufacturer.

Keywords – Hardness tests, Hardness standard blocks, Testing conditions, Variability, and Equivalent indentation depth

1. THE BEGINNING OF HARDNESS STANDARD BLOCKS IN JAPAN

Quantification of hardness, or industrial application of hardness tests, can trace back to the invention of the Brinell hardness in 1900. By the 1920s all of the major hardness testing methods defined in the current JIS standards had appeared. There are records that Brinell and Shore hardness testers were imported to Japan during the 1910s, and domestic production of these testers also started relatively soon thereafter [1-2].

Shoichi Yamamoto, the founder of Yamamoto Scientific Tool Laboratory (YSTL), studied under Dr. Kotaro Honda at Institute for Metals Research of Tohoku University before World War II. Upon a request for domestication of the technology for producing hardness standard blocks, Yamamoto started researches on hardness standard blocks based on the knowledge of steel physics and metallurgy. It was in 1939 when hardness standard blocks were produced domestically for the first time in Japan. After the war, Shoichi Yamamoto and his son Hiroshi Yamamoto established a manufacturer specializing in production of hardness standard blocks in Funabashi, Chiba Prefecture, in 1952 to disseminate the knowledge of hardness standard blocks, as well as to seek higher accuracy of those blocks by the motto: In pursuit of zero variability in hardness values.

As the supply of hardness blocks were getting into full swing in Japan, the Committee for Standardizing Hardness Standard Blocks was set up to resolve the post-war industrial confusion due to inconsistent hardness values. To continue the effort on a nationwide scale, Katasa Kenkvukai (an association for the study of hardness), which is the predecessor of The Material Testing Research Association of Japan, was organized in 1956 under the leadership of Dr. Takeo Yoshizawa, professor of Tokyo University. Through the association, Dr. Yoshizawa gathered the forces of leading researchers and organizations in various fields, including researchers in mechanics (Yamashiro, Kuroki, etc.), researchers in material science (Owaku, Terasawa, etc.), Tokyo Metropolitan Industry Promotion Center, Central Metrological Inspection Center, Nippon Kaiji Kyokai(ship classification society, NK), and manufacturers of hardness testers, such as Akashi (currently Mitsutoyo) and Shimazu. The association made repeated investigations using YSTL hardness blocks on the status of variability in hardness values in various industries, including steel copper rolling, stainless, mill roll, and making, communications, to figure out practical solutions. These efforts led to the establishment of three independent standards for hardness tests earlier than any other nations: for the test method, the testing machine and the standard block. These achievements are compiled into a book titled Hardness Testing Methods and their Applications, which is a must for people concerned in the hardness industry [3-4].



Fig. 1 Demand for Hardness Blocks by Hardness Scale

2. THE PRESENT STATUS OF HARDNESS STANDARD BLOCKS

Today, hardness standard blocks are available in around 140 varieties as shown in Table 1, and their annual output is about 30,000 pieces. Figure 1 shows major hardness scales in demand, including Rockwell, Brinell, Vickers, Micro Vickers, and Shore. Test forces covered by these blocks range from 3,000 x 9.8N to 0.001 x 9.8N as shown in Table 1.

These hardness standard blocks are manufactured and calibrated in accordance with the relevant JIS standards to ensure high uniformity and stability in hardness, and high reliability of hardness standard values.

2.1 Hardness Uniformity

The hardness uniformity of hardness standard blocks is the most important factor that influences the certainty of standard hardness values, which is mentioned later. Therefore, the standard blocks are made from materials of the highest possible quality and in stable supply. Steel and copper alloy, which are often used as the material of hardness standard blocks, must take the form of a flat plate to prevent hardness uniformity from being impaired due to central segregation. The surface of the plate material becomes the test surface of a standard block. A finished block takes the form of a disc, as shown in Figure 2, so that the corner effects caused by heat treatment can be minimized.

Uniformity of the heat-treated microstructure of a hardness block is ensured by choosing the type of material and heat treatment conditions that enable the optimal



Fig. 2 Testing Positions on a Hardness Standard Block



Fig. 3 Quenched Microstructure of a Hardness Standard Block Made of Eutectoid Carbon Steel

microstructure for each testing method, as the dimensions of an indentation can vary by a factor of 1,000 among different testing methods. Eutectoid carbon steel is the most familiar type of steel used as the material for hardness

Assortment	Hardness value	Tolerance	Calibration number(n)	Variation (R=MaxMin)	Materials (JIS notation)	Dimention (mm)	Finished surface	Standard based
HMV (1, 0.1)	1650	±10%	4 (2×2)	2%(HV1)	Si ₃ N ₄	□10×5		JIS B 7735
HMV (1,0.1,0.01)	900, 800, 700 , 600, 500	±15	6 (3×2)	5% (HV0.1)	SK85	¢25×5 ≫		JIS B 7735
*	400, 300, 200 (Be Copper)	±15	11	4	C1720P	\$ 25×6(2)		4
#	100(c2600P), 40(c1020P),	±10	4	7(100HV0.1), 4(40HV0.1)	←	φ25×5 ₩		11
HMV (0.1,0.01,0.001)	30(AU)	±10	"	4 (HV0.1)	Au	¢25×5(0.8)		4
UMV (0.01, 0.002)	900, 700 (Berkovich 9.8mN tested)	±20%	6 (3×2)	10% (HV0.01)	SK85	¢25×6	JIS E	3 7735 *Coming
	500, 200 (*)	*	*		SK85,C1720P	¢25×6(2)		*
HV (30, 1)	1000(sk120), 900, 800, 700	±15]	10(5×2)/HV30,10	1.5%	SKS3	¢64×15	0	JIS B 7735
HV (10,1)	600, 500, 400, 300, 200 (SK85), 150 (S45c)		6(3×2)/HV1	4	←	*	0	#
4	100(C2600P), 40(C1020P)	±10]		(150HV and below 2.2%)	←	φ64×10	0	#
HS	100(SK120), 95, 90, 80, 70, 60, 50, 40, 30	±2	HV10(5×2)	$R~VHS {\cong} 1.5 \mbox{(70HS and below 1.2)}$	SK85	¢64×15	0	JIS B 7731
*	20 (S20C), 7(C1020P # 64×10)	* Ĵ	HS10(5×2)	Δ HS(HS-VHS) \leq 0.5	←	4	0	4
HL	HLE (Dia) 850, 800, 700, 600, 500	$\left. \stackrel{\pm 15}{*} \right\}$ (HV Calibratio	(IN) O-Therefore)		SK85	¢115×33	Ö	JIS B 7731
	HLD(wc)880, 830, 730, 630, 520		(HV Calibration)	1	1	4	0	Related
HR C	70(SK120), 67, 64, 62, 60	±1	10(5×2)	0.2	SKS3	¢64×15	0	JIS B 7730
"	57, 55, 50, 45, 40, 35, 30, 25, 20, 10	*	1	(40RC and below 0.3)	SK85	*	0	4
HR A	87, 85, 83, 81, 78, 75, 71, 65, 56	4	*	0.3	Same as HRC	4	0	"
HR 30N	83, 81, 78, 73, 67, 60, 55, 50, 41	4	4	0.6	4	4	0	4
HR 15N (45N)	92, 90, 87, 85, 80, 75 (43) (23)	"	"		4	"	0	4
HR B	100, 95, 90	±2	10 (5×2)	0.8	SK05	∉64×10	Δ	JIS B 7730
"	82, 72, 62, 52, 42, 32		4	(50BB and below 1.0)	C2600P		Δ	4
HB 30 T	78, 72, 62, 52, 42, 38, 32	4		1.0	Same as HRB	4	0	4
HR 15 T	87, 82, 78	4	*	4	*	4	0	4
HB(E-M-L-B-F-S)	HBE90 HBM107 HBL118 HBB123 HBE9	0 /	4	4	(100HV)	4	0	JIS K 7202
*	HBM 67 HRL92 HRR105 HRS9	0 *	"	4	(40HV)	"	Ō	#
HBW 10/3000	600, 550, 500, 450, 400, 350	±15	6 (3×2)	1.5%	SK85	¢115×18	•	JIS B 7736
HB10/3000	300, 250, 229 (d=4mm), 200, 180	4	4	4	4		٠	*
	HB (10/3000) 150, HB (10/500) 125	4	1	2.5%	S45C	4	•	*
	HB (10/500) 100	4	"	3%	S10C	4	÷	4

Table 1. Specifications of Hardness Standard Blocks

Finished test surface; ●Fine grinding, △plate lapping, ○Buffing, □Super finish, ■Super finish(fine), New Test Blocks (N. T. B.)%(for Spot Anvil) ◇Export only %¢25×6

standard blocks. This material requires strict control over its quenching conditions, but the quenched microstructure does not include excessive carbide, enabling a hardness block to achieve high hardness uniformity even in the micro-hardness testing range.

2.2 Reliability of Hardness Standard Values

The value indicated on a hardness standard block must be a representative value for hardness measurements available through industrial hardness tests. Therefore, arbitrary testing conditions should be avoided when determining the standard value indicated on a hardness block. There should be no conflicts between the testing—load applying—method generally used in the industries and the standardized testing method for determining the standard value indicated on hardness blocks.

At YSTL, all hardness blocks are produced on a lot-by-lot basis throughout the entire production process until they are tested to determine standard hardness values. One lot consists of 20 blocks, as shown in Figure 4. The testing for determining standard values consists of two sets of taking measurements at five points as shown in Figure 2 to check for differences among different testing machines. Rockwell hardness tests tend to develop differences in hardness measurements among indenters. Therefore, the average of hardness measurements with about 170 diamond indenters is taken to determine the standard value. Figure 5 shows a comparison of HRC, HRA and HRD hardness measurements with 30 of the said diamond indenters.

The results of testing all hardness standard blocks are recorded in data sheets, which are stored in their original writing and electronic forms. The first-numbered block is not for sale, and stored for warranty purposes. The warranty period of a hardness standard block is three years from the date stamped on the attached inspection tag. During the



Fig. 4 20 Hardness Standard Blocks Per Lot

warranty period, we offer services, such as providing data, retesting the warranty block, and carrying out microscopic examinations upon user requests.

2.3 Hardness Stability

To prevent changes in the properties of hardness block material, subzero treatment is provided to steel blocks, immediately after the quenching process, according to the level of hardness, to minimize residual austenitic structure within quenched martensite. For low-hardness standard blocks often made of copper alloy, the optimal surface treatment method is applied, and stress relief annealing is provided as necessary to minimize the residual working stress arising from the test surface finishing process. These efforts are made to warrant that a hardness standard block does not produce changes in hardness exceeding the resolution of a hardness testing machine during the warranty period. To achieve that purpose, it is important to keep the warranty standard block in good condition.



Fig. 5 Comparison of Hardness Measurements with Different Rockwell Diamond Indenters

3. STUDIES ON LOAD APPLYING CONDITIONS

Once the uniformity and stability of hardness is ensured for hardness standard blocks based on metallurgic studies of hardness block material, you should then look at the testing conditions for determining standard values. There used to be a discrepancy in testing cycle, or load applying conditions, between that defined in the JIS standards for the hardness test method [5] and for the standard blocks [6]. Therefore, even if the JIS-compliant testing cycle is followed using a testing machine and indenter whose direct accuracies are verified, it could not be expected that the results of hardness measurement agreed with the values indicated on hardness standard blocks.

To address this issue, the authors made continuous studies on load applying conditions using high-accuracy hardness standard blocks. As a result, the current JIS standards for hardness standard blocks adopt the testing cycle that is consistent with that defined in the standards for the hardness test method.

Figure 6 shows hardness measurements as a function of indentation velocity. LRT in the figure stands for Load Rise Time—a period until the total test force is reached Figure 7 shows hardness measurements as a function of load duration. time—a period during which the total test force is maintained.

Even after the discrepancy in testing cycle was eliminated, there remained a difference in the way of defining the loading velocity: LRT was adopted by the industrial side, whereas indenter velocity was used by the metrological side. To solve this problem, the authors analyzed the issue from the viewpoint of strain velocity, and revealed that, given that application of the test force *P* is shown as a function of time (t) with the equation $P(t) = a \times t^n$, the strain velocity is determined by LRT, as shown in Equation (1). This provided substantiation for the reasonability and convenience of using LRT to define the loading velocity for hardness testing.

$$\dot{\varepsilon}(t) = \frac{1}{2} \alpha \frac{n}{t} \tag{1}$$

On a practical basis, it is now proved that the LRT alone is sufficient to define the loading velocity for hardness testing [7].

4. Other Sources of Variance in Hardness Measurements

The loading conditions described above are a representative source of variance in hardness measurements, but there are other sources to be addressed at the level of hardness block manufacturer.

4.1 Test Force Error

When a test load of varying forces is applied to a test specimen of uniform hardness like a hardness standard block, the size of the resulting indentations is approximately proportional to the square root of the test load. However, if it is let unnoticed that the variance in size of the



Fig. 6 Hardness Measurements as a Function of Indentation Velocity



Fig. 7 Hardness Measurements as a Function of Load Duration Time

indentations is attributable to test force errors, such variance would be regarded as entirely coming from errors of hardness measurements, which is a problem that tends to happen at the site.

Although test force errors are strictly defined [8-9], the accuracy of a test force is verified statically, which does not necessarily reflect the accuracy of dynamic test load being applied. The static verification of the test load accuracy is often done using an elastic proving device. The proving device is mounted on a hardness testing machine, after the indenter and the specimen are removed from the machine, and applied the test force to take measurements of the force upon being stabilized. Actually, however, it is the dynamic test force that influences the size of indentations. It is therefore recommended to establish a method for dynamically verifying the test force accuracy, and provide a numerically more moderate, but effective, definition of test force errors, instead of demanding unnecessarily strict control over static test force errors.

Seemingly, the current standards for Rockwell hardness testing machines and standard blocks excessively require high accuracy for both of the 1st and the 2nd preliminary forces before the total test force is applied and after the total test force is removed, respectively. That only seems to increase costs and technical difficulties for producing the testing machines, considering the considerably smaller effect of the 2nd preliminary force error on the variance of hardness measurements, compared with that of the preliminary force error, as evident from Figure 8 [10] It should also be noted that the above-mentioned standards only define static test force errors, not dynamic ones—which actually have more influence on the variance of hardness measurements.

4.2 Testing Temperature

It is a well-known fact that the hardness of a hardness standard block itself, not the hardness measurements taken on the block, can vary with temperature. It is also expected that changes in the performance of hardness testing machines due to temperature differ from one model to another. The authors investigated the influence of changing temperatures on hardness measurements via two Rockwell hardness testers at and around room temperature by mounting thermocouples onto the standard blocks, the anvils and the diamond indenters used. The result is shown in Figure 9, which suggests that the ambient temperature does have a slight effect on hardness measurements for determining the standard value of hardness standard blocks.

However, considering that not a few hardness testers have built-in motors, computers and lighting equipment the exhaust heat from which can influence hardness measurements, strict control over the temperatures of the testing room and the standard block alone is not very meaningful. In addition, because a diamond indenter is a good conductor of heat, the temperature of the indenter can differ from that of the room and the standard block. In that case, the area where the diamond tip contacts the block would experience a rapid temperature change. Therefore, it is almost meaningless to require stricter temperature control than usually expected from a common-sense viewpoint, and the current level of requirement for temperature control specified in the standards for the Rockwell hardness testing method and standard blocks seems to be practical enough [8-9].

4.3 Number of Indentations over a Standard Block

The influence of the number of indentations made on a standard block on hardness measurements becomes an issue, particularly for Rockwell hardness. As shown in Figure 10, hardness measurements become higher as the number of indentations increases, and such trend is more evident with softer hardness blocks. To address this issue, ISO once discussed revising its standard to increase the thickness of a standard block for soft metals, such as the Rockwell B scale. However, a plate metallic material with a low rate of rolling reduction tends to inhibit miniaturization of grain size [11] and hardness uniformity. Figure 10 also indicates that even the 70 HRC steel block, which is much harder and thicker than a copper alloy block, does develop a change in hardness measurements with the number of indentations. Accordingly, the authors think it unnecessary to increase the thickness of Rockwell hardness blocks currently specified in the JIS standards, and that a thicker block would have adverse effects, such as lower hardness uniformity and restrictions on manufacturing and handling [6],[9].







Fig. 9 Hardness Measurements as a Function of Temperature on 60 HRC Steel Blocks

5. STANDARD BLOCKS IN RELATION TO THE LATEST HARDNESS TESTING TECHNOLOGIES

As mentioned earlier, the currently standardized hardness tests were mostly invented before World War II. Since the end of the war, there has been a considerable progress in researches about micro hardness testing methods, primarily for Vickers hardness. A new repulsive hardness tester based on the ratio of indenter velocities before and after the impact [12] was also developed. In addition, there were advances in the studies of dynamic hardness testing, with which hardness is determined by measuring the indentation depth under load [13-14]. The studies on dynamic hardness testing have evolved into the instrumented indentation test standardized in ISO 14577-2002 [15-16], which receives increasing attention in Japan as a nanoindentation test for micro hardness.



Fig. 10 Change in Hardness Measurements with an Increase in the Number of Indentations

5.1 Development of Nanoindentation Hardness Blocks

Figure 11 shows the appearance of a hardness standard block made of single crystal tungsten, which has been developed for nanoindentation testing. The pop-in phenomenon—instantaneous transition from elastic to plastic deformation—observed in single crystal tungsten while being indented provides an intuitive picture of the wearing status of the tip of a diamond indenter [17]. This instrumented indentation test satisfies the similarity rule of hardness [18-19], and is available for measuring ultra micro hardness in the nanoindentation range. However, it still requires complicated compensations via various standard test pieces for the elastic deformation of a testing machine frame and the difference from the ideal geometry of the indenter tip.

5.2 New Hardness Method Based on Equivalent Indentation Depth

Using the nanoindentation hardness block, the authors examined some issues to address as regards the instrumented indentation test, and make ongoing efforts to realize practical application of the proposed concept of "equivalent indentation depth." As shown in Equation 2, the equivalent indentation depth is calculated by dividing the depth of indentation Δh —which is obtained using the preliminary test forces as with the Rockwell hardness testing, as indicated in Figure 12—by the square root of the total test force. This provides an indicator of indentation depth not subject to the test force applied. For this indentation testing, an indenter of the shape to which the similarity rule of hardness applies, such as Vickers indenters, should be used.

$$\Delta h_{\rho} = \Delta h / \sqrt{P} \qquad (2)$$

Figure 13 shows the values of Δh_e obtained by testing on



Fig. 11 Hardness Standard Blocks Made of Single Crystal Tungsten for nano indentation

metallic standard blocks of 100 HV to 1,000 HV under several test forces. The values of Δh_e are in good agreement between the different test forces [20].

6. REVIEW OF THE ISO STANDARDS FOR HARDNESS

The above discussions are summarized below to present opinions concerning the ISO standards for hardness in the capacity of a hardness block manufacturer.

(1) Given the impact of the ISO standards to other industrial standards, such as JIS, simplification, particularly of ISO 14577, is desired to facilitate practical application, while with due respect for the principles of the standards.

(2) It is desired that some mandatory practices of which the idea is right but practical exercise is difficult, such as the practice of direct verification when installing a new testing machine, should be changed into recommended practices.

(3) No matter how thick a hardness block is, an increasing number of indentations do cause variance in

hardness measurements. Specifying unnecessarily large thickness for standard blocks for Rockwell and other scales, would only make it difficult to use high-quality rolled material, especially for copper alloy blocks, resulting in lower hardness uniformity of the blocks.

(4) Because it is difficult to take precise dimensional measurements of an indentation under the loading range for micro Vickers testing, the ISO seems to rely on the workaround of increasing the number of indentations to be measured to secure the reliability of the standard values of hardness standard blocks [21]. However, the reliability of the standard values may not be increased, no matter how many measurements are taken of poorly defined images of indentations. Therefore, it is desired to provide a more realistic measure, such as allowing larger tolerances.

(5) As to hardness testing temperatures, the ISO's current requirement of being controlled at 23°C±5°C should be left unchanged. Stricter requirement would only make it meaningless, considering the actual status of testing machines and the testing environments.

(6) The current same level of strict tolerances for both the 1st and the 2nd preliminary test forces for Rockwell hardness testing only discourages industrial application of the ISO standards. It is highly recommended to accept larger tolerance for the 2nd preliminary test force.

(7) As to test force efforts, it is recommended to establish a method for dynamically verifying the test force accuracy, and to introduce a numerically more moderate definition of test force errors accordingly.

(8) The loading condition, i.e. test cycle for indentation hardness tests should be generally defined by length of time

7. CONCLUSION

The history of hardness tests has been in parallel with the development of materials and their treatment technologies. Hardness testing methods that are currently available for extensive industrial applications are all developed for the original purpose of measuring the strength of metallic materials. Metallographic examination and hardness testing are the first and foremost ways of testing materials, and as such they play an important role for the safety of industrialized society.

In too much recognition of such importance, however, there seems to be excessive pursuit of stricter requirements as to uncertainties of hardness measurements, traceability, and formalities such as certificates of calibration. The original purpose of hardness tests should not be neglected. Even if you are familiar with such certificates or uncertainties, lack in the ability to provide daily maintenance of hardness testers and related instruments and/or the knowledge of the basics of hardness testing methods would make it difficult to serve for the safety of industrialized society or might impair it.

Hardness tests are supposed to be used at the discretion of those who carry out them according to their respective purposes. This freedom drives the technical development of these tests. Meanwhile, if business transactions require a comparison of hardness measurements of materials, including standard blocks, industrially unifiable practical



Fig. 12 Measuring the Indentation Depth Used for Calculation of the Equivalent Indentation Depth





standards should be provided to enable the comparison.

Being back to the basics of hardness, the measurements of indentation hardness rely mostly on the factors of "force" and "length": the test force applied and the dimensions of an indentation. Therefore, the accuracy of hardness is primarily subject to that of force and length, and secondarily to time, or loading condition, i.e. test cycle and the testing temperature. No strict argument over the digit number of measurements is heard for other measures of material strength than hardness. The pursuit of too much strictness in hardness measurement might produce opposite effects.

Standard blocks for hardness are produced to be extremely uniform and stable in hardness by using the highest-quality materials and careful treatment procedures. Such blocks are tested with one of the most reliable testing machines in the industrial world, under the testing conditions that are most widely adopted, to determine the hardness standard values to be indicated on the blocks. As such the standard blocks should qualify as standard test pieces for hardness measurement.

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