

DEFINITION OF NEW INDUSTRIAL HARDNESS TEST USING EQUIVALENT INDENTATION DEPTH

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Abstract – The Rockwell hardness test is commonly used and accepted by many industrial users, but the hardness value requires conversion between scales because the geometry of the indenters and the load ranges are different. On the other hand, hardness calculation using pyramidal indenters is quite simple and can be applied to any load range, though it is much more complicated to calibrate the frame compliance and the truncation of indenters in nanoindentation measurement. For this reason, nanoindentation measurement is not yet fully industrially friendly.

In order to improve the above situation, newly developed industrial hardness test methods and a new concept of “Equivalent indentation depth (or indentation depth index)” were proposed and investigated in this paper. These methods are based on the principle of “similarity” of Vickers and other pyramidal indenters, and take advantage of the industrial and practical usability of the Rockwell hardness test. Experimental results that covered macroscopic through nanoscopic ranges show that the methods can be applied to a wide range of test loads. One of the expected advantages of the methods of performing nanoindentation and other instrumented indentation tests (ISO 14577) is that the hardness is not significantly influenced by the frame compliance or the truncation of indenters. Other advantages over conventional hardness tests are also discussed in this paper. More experimental work is necessary to confirm and establish the new methods.

Key words – new hardness test, similarity, indentation depth index, nanoindentation, Rockwell hardness

1. INTRODUCTION

The Rockwell hardness test [1], which is a representative industrial hardness test, is easy to carry out and is industrially the most successful test method. However, because it does not take into account the similarity rule of hardness, in theory different hardness values will be obtained for different loads, even when the same test piece is tested, and a comparison of hardness values between a large number of scales necessitates a hardness conversion table (relation table) [2].

In contrast, other tests such as the Vickers hardness test and the ISO 14577 instrumented indentation test [5] which take account of the “similarity rule of hardness” [3-4] are superior test methods which can theoretically cover macroscopic through nanoscopic ranges as coherent hardness

values. Regarding the former test, however, measurement of the indentation dimension relies on the use of an optical microscope, and there is a technical limitation due to the resolution of the microscope, hence it is difficult to use this method in a nanoscopic range test. The latter test is a revolutionary test method that permits the so-called nanoindentation test to be performed, however in the nanoscopic range there are large errors due to the incompleteness of the material surface detection and the geometry of the spherical tip of the diamond indenter, while in the macroscopic range there are large errors in displacement measurement due to deformation of the tester when a load is applied, necessitating complicated correction [6-7] for each standard material. It is therefore difficult to say that this method can be easily used on an industrial basis.

In this report, the features of these test methods are studied, and the results of basic research related to proposals concerning new industrial hardness test methods to permit unified evaluation of hardness from macroscopic through nanoscopic ranges are set out.

2. DEFINITION OF NEW HARDNESS TEST METHOD

2.1. Measuring the Indentation Dimensions, and Indenters

An industrial hardness testing method must first of all be easy to perform. It must also be capable of clarifying the ranking of hardness or softness between different materials, based on a constant definition. Also, to enable the test method to perform a coherent evaluation from macroscopic through nanoscopic ranges, it is necessary that the principle of measurement satisfy the similarity rule of hardness.

In order to simultaneously satisfy these two points, a test method which uses reference load as in the Rockwell hardness test, and also uses an indenter that satisfies the similarity rule of hardness as in the Vickers or Berkovich diamond indenter, is proposed as a definition of a new industrial hardness test method.

2.2. Equivalent Indentation Depth Based on the Similarity Rule of Hardness

Generally, when attempting to define an industrial hardness value or a hardness ranking using the indentation depth h , if the industrial hardness is defined as H_E , it is possible to define the relationship $H_E = F(h)$. For example, the well-known Rockwell hardness is defined as a linear function of h .

According to Tabor, if the test piece is ideally uniform, the indentation due to a pyramidal indenter or a conical indenter for which the similarity rule of hardness holds enables the same theoretical hardness to be obtained even if the indentation dimensions vary with the size of the load, because the geometrical shapes of the indentations are similar. In other words, if the theoretical hardness value is expressed as H_T , the relationship " $H_T = P/A_P = C \times P/h^2 = \text{const}$ " holds (where, P : Testing load, A_P : projected area of the indentation, C : Constant determined by the geometry of the indenter), hence \sqrt{P}/h remains constant regardless of the size of the testing load P .

The former industrial hardness H_E is a function of h alone, so it is not possible to satisfy the similarity rule of hardness regardless of the indenter used. However, if instead of h the equivalent indentation depth (indentation depth index) h_e is defined by $h_e = h/\sqrt{P}$ as an index of the indenter depth which does not rely on the size of the testing load P , the relationship $H_E = F(h_e)$ will hold, and it will be possible to define a coherent hardness value that does not depend upon the size of the testing load.

2.3. Measurement of the Equivalent Indentation Depth Using the reference Load Method

In the Rockwell hardness test, first the reference load P_0 is applied to the indenter, causing it to be pushed into the test piece. The position of the indenter at this time is defined as the origin. Next, the testing load P is applied, then the load is reduced to P_0 , and the difference Δh between the indenter depth at this time and the origin is measured.

Kuroki and Mashimo independently carried out research on Rockwell hardness spherical-conical diamond indenters and testing load, and proposed improvements to the Rockwell hardness test method. However, because of technical restrictions existing at the time the research was carried out, the testing load was limited, and in principle the test method was not capable of freely changing the load and satisfying the similarity rule of hardness. [8-10]

On the other hand, by using a pyramidal or conical diamond indenter, it can be expected that due to the similarity rule of hardness the indentation geometry will be a similar

type that is proportional to \sqrt{P} . Consequently, if this concept of equivalent indentation depth is applied to the calculation of the abovementioned Δh as well, the following relationship will hold.

$$\Delta h_e = \Delta h / \sqrt{P} \quad (1)$$

It is thus possible to satisfy the similarity rule of hardness without being restricted to the size of the load P . However, Δh exists not only in the testing load P but also in the reference load P_0 as well, so in order to perform a hardness test while freely changing the testing load, it is necessary to maintain the ratio of P_0 and P constant, as expressed by the following equation.

$$r = P_0/P = \text{const.} \quad (2)$$

If the test is performed according to conditions (1) and (2), in principle Δh_e will be constant for all values of testing load P from macroscopic through nanoscopic ranges. Consequently, in the following equation, the hardness value will also be constant, regardless of the loads.

$$H_E = F(\Delta h_e) \quad (3)$$

3. TEST METHOD

In order to study the appropriateness of the above principle, the Vickers hardness standard block shown in Table 1 was subjected to a macroscopic test using a Rockwell hardness tester and also a Rockwell superficial hardness tester, and a proving test of the principle of the equivalent indentation was carried out. Also, taking into consideration the possibility of applying this principle to the nanoscopic range in the near future, it was decided to perform a nanoscopic range test on the micro Vickers hardness standard block for reference using an instrumented indentation tester (nanoindentation tester).

3.1. Tester and Indenter

Regarding the macroscopic range test, two Vickers

Table 1 Standard blocks for hardness used as specimens

Scale	Hardness	Dimension (mm)	Material (JIS)	Microstructure
Vickers H V	979 HV30	$\Phi 64 \times t15$	Steel (SK120)	Martensite
	905 HV30		Steel (SKS3)	Tempered martensite
	702 HV30			
	507 HV10		Steel (SK85)	Troostite
	293 HV10			Sorbite
	158 HV10		Steel (S45C)	Ferrite and Cementite
103 HV10	$\Phi 64 \times t10$	Brass (C2600P)	Annealed structure	
Micro-Vickers HMV	1652 HV0.1 (1616 HV1)	$\square 10 \times t5$	Si_3N_4	
	902 HV0.01	$\Phi 25 \times t6$	Steel (SK85)	Tempered martensite
	698 HV0.01			Troostite
	506 HV0.01			Sorbite
	304 HV0.01			
102 HV0.01	Brass (C2600P)	Annealed structure		

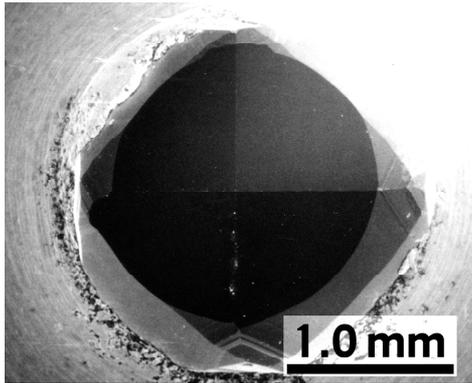


Fig. 1 SEM image of Vickers diamond indenter for macro range test

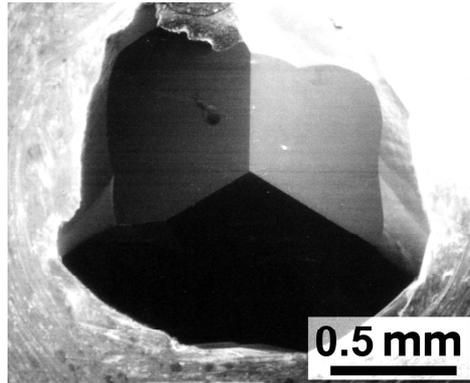


Fig. 2 SEM image of Berkovich diamond indenter for nano range test

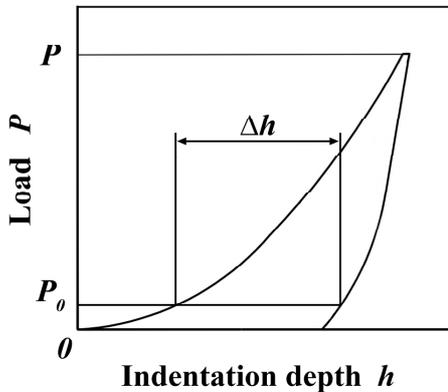


Fig. 3 Explanatory diagram of Δh obtained by indentation test

diamond indenters were installed on Akashi Wizard HR522 and Wilson 2001 T Rockwell hardness testers and also on Akashi Seisakusho ORK (S) and AHT (S) Rockwell superficial hardness testers, and tested. The size to which the diamond of each of these indenters was finished was approximately 1.5 mm along the diagonal, as shown in Fig.1. Consequently, the test was carried out using a combination of a testing load and test piece hardness that produces an indentation of no greater than this.

Regarding the reference test in the nanoscopic range, a dedicated Berkovich diamond indenter was used on an Elionix ENT-2100 and also on an MTS DCM nanoindentation tester and tested.

As indicated in Fig.1 and Fig.2, the Vickers and Berkovich diamond indenters are pyramidal and conical, respectively. However, the relationship between the surface area A and the depth h is designed to be the same for both types of indenters, i.e. $A = 26.43/h^2$. Also, it is known that the hardness values obtained from the indentation areas match each other relatively well. [11-12], [6]

3.2. Test Procedure and Calculation of Δh_e

In the case of the Rockwell hardness tester and the Rockwell superficial hardness tester, Δh was reverse-calculated from the value displayed on the tester in

accordance with the Rockwell hardness definition equation¹⁾. In the case of a nanoindentation tester, Δh was obtained as shown in Fig.3 from the numeric load-displacement data continuously taken into a computer. In both cases, Δh_e was calculated using Eq. (1).

It was decided to fix the ratio r of P_0/P to $r = P_0/P = 1/15$ and also $1/10$ for the convenience of using the Rockwell hardness tester and also the Rockwell superficial hardness tester. The testing load P in the case where $r = 1/15$ was set to 150×9.8 (N) for using the Rockwell hardness tester, and was set to 45×9.8 (N) for using the Rockwell superficial hardness tester. Also, the testing load in the case where $r = 1/10$ was set to 100×9.8 (N) for using the Rockwell hardness tester, and was set to 30×9.8 (N) for using the Rockwell superficial hardness tester. In the case of the nanoindentation tester, the testing load was set to 0.0015×9.8 (N) irrespective of the value of r .

3.3. Loading Conditions

In order to carry out tests at the same strain speed from the nanoscopic range to the macroscopic range, and accurately establish the similarity rule of hardness, it is advisable to use constant loading conditions regardless of the size of the testing load. [13] In this research, however, regarding the Rockwell hardness tester and the Rockwell superficial hardness tester used for macroscopic range testing, the load application conditions were made to conform to the ISO 6508 “Metallic Materials - Rockwell Hardness Test” standard [1], and concretely the “additional load application time” was made approximately 3 s, and the “total load dwell time” was made approximately 5 s.

Also, regarding the reference test performed with an instrumented indentation tester used in the nanoscopic range, tests were performed using the manufacturer’s recommended conditions.

3.4. Number of Measurement Points

In the macroscopic range, regarding the Vickers hardness standard blocks shown in Table 1, measurements were performed at five points for each combination of tester and diamond indenter. Consequently, one hardness standard block was measured using two Rockwell hardness testers and two Rockwell superficial hardness testers, and two indenters,

at five points each, that is, a total of 20 points, for each testing load.

In the nanoscopic range, a micro Vickers hardness standard block was measured at a testing load of 0.0015×9.8 (N) using two testers, at five points each. Also, an HMV 1600 silicon nitride ceramic micro Vickers hardness standard block was tested under the abovementioned testing load of 0.0015×9.8 (N), and was also tested at 45×9.8 and 30×9.8 (N) for reference.

4. TEST RESULTS

Table 2 shows Δh when a Vickers hardness standard block is measured using a Vickers diamond indenter under a testing load in the macroscopic range, and also the equivalent indentation depth Δh_e calculated from it, separately for $r = P_0/P = 1/15$ and $1/10$. Figure 4 shows a plot of Δh_e at 150×9.8 (N) with respect to Δh_e at 45×9.8 (N) for $r = 1/15$, and also a plot of Δh_e at 100×9.8 (N) with respect to Δh_e at 30×9.8 (N) for $r = 1/10$, in order to compare the equivalent indentation depths for different testing loads.

Looking at the results for the macroscopic range, it can be seen that the difference between the values of Δh_e for testing loads of 150×9.8 and 45×9.8 (N) for $r = 1/15$ is no more than about 1%, which indicates good agreement, and the difference between the values of Δh_e for testing loads of 100×9.8 and 30×9.8 (N) for $r = 1/10$ is a similar value. Consequently, it can be considered that the existence of a similarity rule of hardness obtained using an equivalent indentation has been proved.

On the other hand, concerning the test results for the nanoscopic range, although both the indenter geometry and the test piece are different from those of the macroscopic range, the results of comparing these test results with those for a test load in the macroscopic area of between 20,000-fold and 100,000-fold are shown for reference in Fig.5. The variation in the test results in the nanoscopic range is large, and it is difficult to make an accurate comparison, so it is necessary to carry out further studies in the future.

5. DISCUSSION

Based on the foregoing proving test results, the results of a study concerning the applicability of the equivalent indentation depth to new industrial hardness test methods are set out below.

5.1. Features of New Industrial Hardness Test Method Using Equivalent Indentation Depth

(1) This is an industrial test method which uses reference load like Rockwell hardness. It has been devised so as to satisfy the similarity rule of hardness, so even if the test load is changed the hardness can be evaluated coherently as the same hardness.

(2) Because the test involves measuring the indentation depth, it can be performed in a shorter time compared to the measurement of the indentation dimension using a microscope.

(3) Because a reference load is used, frame compliance of the tester virtually does not constitute a problem, and there is no necessity to perform correction or calibration for each type of hardness standard block.

(4) Because the reference load is used at the origin of indenter depth measurement, the effect of errors with respect to the ideal geometry of the indenter tip is small, and it is predicted that errors in origin detection do not readily occur.

(5) Basically, this industrial hardness test is the same as Rockwell, Shore, Leeb, and other hardness tests, so it is not suitable for comparing the tensile strength or yield strength of materials.

(6) Compared to an instrumented indentation test, it is considered that this test is somewhat unsuitable for the evaluation of materials that have large elastic recovery of indentation.

(7) The upper limit of the load in the macroscopic range must be approximately that of the Rockwell hardness tester (max 1471 N) because of the indenter manufacturing limitations.

5.2. Definition of Hardness Value due to Equivalent Indentation Depth

Table 2 Equivalent indentation depth Δh_e by Vickers diamond indenters at $r = 1/15$ and $1/10$

Standard blocks	$r = 1/15$				$r = 1/10$			
	Δh (μm)		Δh_e		Δh (μm)		Δh_e	
	150 ($\times 9.8$ N)	45 ($\times 9.8$ N)	150 ($\times 9.8$ N)	45 ($\times 9.8$ N)	100 ($\times 9.8$ N)	30 ($\times 9.8$ N)	100 ($\times 9.8$ N)	30 ($\times 9.8$ N)
1616 HV1		15.3		2.28	11.7			2.13
979 HV30	42.4	23.2	3.46	3.46	31.9	17.4	3.19	3.18
905 HV30	45.5	24.9	3.71	3.71	34.1	18.7	3.41	3.41
702 HV30	54.4	29.7	4.44	4.43	41.0	22.4	4.10	4.08
507 HV10	66.3	36.2	5.42	5.39	50.0	27.3	5.00	4.99
293 HV10	90.0	49.1	7.35	7.32	67.9	37.2	6.79	6.79
158 HV10	126	68.7	10.3	10.2	94.8	51.8	9.48	9.45
103 HV10		87.0		13.0	121	65.2	12.1	11.9
39 HV10					117			21.3

Notice : Δh_e are calculated as $\Delta h_e = \Delta h / \sqrt{P/9.8}$ in this table.

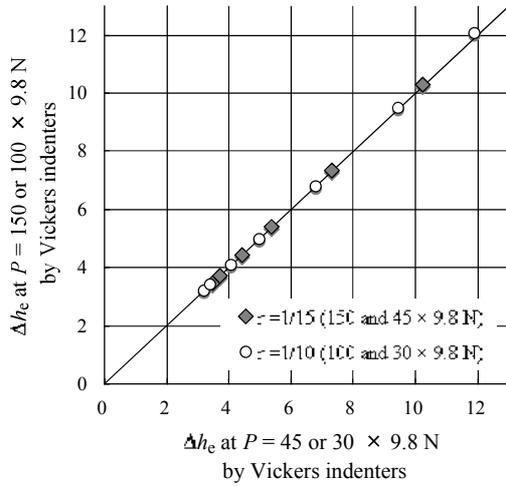


Fig. 4 Comparison of Δh_e between different macro range testing load

As shown in Fig.6, for defining an industrial hardness value using an equivalent indentation depth it is possible to use methods such as Type A in which the indentation depth is multiplied by constant K_2 and the result subtracted from constant K_1 (Rockwell hardness method), and Type B in which constant K_2 is multiplied by the inverse of the indentation depth. It is also possible to use Type C in which the inverse of the indentation depth is squared and displayed as a value multiplied by constant K_2 on the hardness tester. It is considered necessary to carry out careful studies, and define the most appropriate hardness calculation equation from an industrial viewpoint.

5.3. Conversions between r

It is clear from the definition shown in Fig.3 that for each different r , the equivalent indentation depth Δh_e is different. In the case where the indentation depth throughout all stages of a test is recorded, this is not a problem because it is possible to change r to an arbitrary value even after the test. However, in the case of a tester, such as a Rockwell tester, in which the reference load must be set in advance, the dependency of equivalent indentation depth on r makes it inconvenient in practical application.

Accordingly, here a simple model was used and an examination of the method of converting the results for a certain r into the results for a different r was performed.

(1) Contents of modeling

Regarding the relationship between load P and the indentation depth h , and also regarding the loading process and the unloading process, it was assumed that equations (4) and (5) held approximately.

$$P = a h^2 \quad (4)$$

$$P = b (h - h_f)^2 \quad (5)$$

a and b are constants, and h_f is the indentation depth when the load is completely removed. Although these equations are simple approximations, as is indicated later they provide reasonable good results as primary approximations.

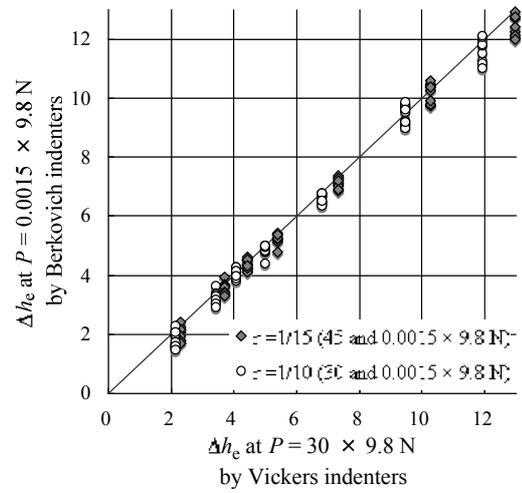


Fig. 5 Comparison of Δh_e between macro and nano range testing load

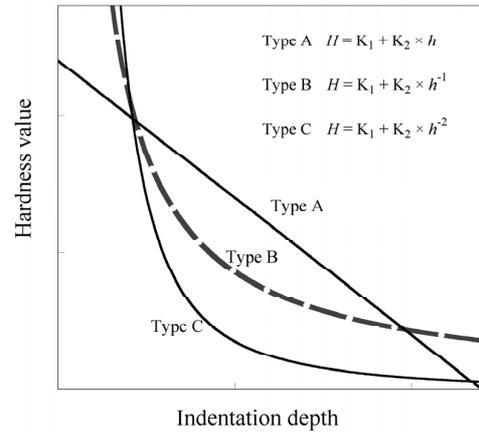


Fig. 6 Relationship between industrial hardness values and indentation

(2) Deriving a conversion equation

If the maximum indentation depth at total load P is defined as h_{max} , and the indentation depths when the reference load $P_0 (=rP)$ is applied and also when it is removed are h_1 and h_2 , respectively, the following equations will hold from equations (4) and (5).

$$\begin{aligned} P &= a h_{max}^2 \\ P &= b (h_{max} - h_f)^2 \\ rP &= a h_1^2 \\ rP &= b (h_2 - h_f)^2 \\ \Delta h &= h_2 - h_1 \end{aligned}$$

These equations are solved to yield the following equation.

$$\Delta h = (1 - r^{1/2}) h_f \quad (6)$$

Because h_f does not rely on r , the formula used to convert Δh ($\Delta h_A, \Delta h_B$) of different r (r_A, r_B) is as follows:

$$\Delta h_B = (1 - r_B^{1/2}) / (1 - r_A^{1/2}) \times \Delta h_A \quad (7)$$

For example, in order to convert the result of Δh of $r = 1/15$ into $r = 1/10$ for the same maximum load, Δh is multiplied by 0.9218, and in the reverse case Δh is divided by 0.9218. Regarding Δh_e as well, conversion can be performed in exactly the same way.

(3) Application to the test results

Figure 7 shows the results of using Eq. (7) to compare the results of $r = 1/15$ and $r = 1/10$. Before the conversion, the values deviate from a 1:1 relationship, however by using Eq. (7) to multiply the results of $r = 1/15$ by 0.9218 and plotting them, the results of $r = 1/15$ and $r = 1/10$ lie on a 1:1 line, and it can be recognized that the model used in this examination is effective to a certain extent.

The effective range and accuracy of this model and also points of improvement will be examined from now on as well. In the case where the model is recognized as having a certain degree of practicality, it can be expected that the convenience to the user can be increased by defining the effect of r in advance in a form in which it is included in an equation for calculating the hardness.

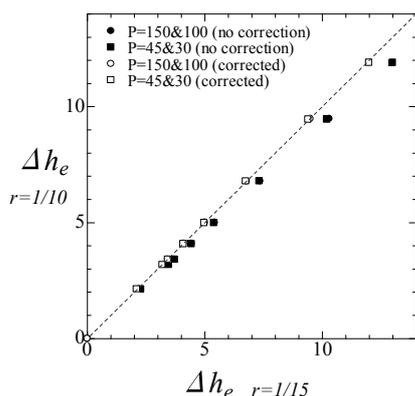


Fig. 7 Comparison of Δh_e between $r = 1/15$ and $r = 1/10$.

6. CONCLUSION

No almighty hardness test method that is suitable for all materials, hardness values and test pieces exists. For example, the ISO 14577 instrumented indentation test on which expectations are being placed as a unified test method from the macroscopic range to the nanoscopic range operates on an excellent principle, however it appears that the complicated correction methods and standards have kept this test method away from industrial use. In addition, the results of current research concerning new industrial hardness test methods proposed for solving problems concerning conventional test methods are summarized below.

(1) By developing the principle of the Rockwell hardness test, making P_0/P , which is the ratio of the reference load P_0 to the testing load P , constant, multiplying the depth

measurement value Δh by $1/\sqrt{P}$, and using an index of indentation depth, i.e., equivalent indentation depth, it became possible to satisfy the similarity rule of hardness.

(2) From the equivalent indentation depth, it is possible to provide industry with new unified industrial hardness values.

(3) By improving this hardness test method, it will no longer be necessary to correct the frame compliance of the tester, and also it is considered that it will help reduce the disparity between measured hardness values attendant to test piece surface detection errors.

(4) Although it is not possible to completely eliminate the effect of a geometry error in the tip of the indenter, it is expected that there will be a significant improvement compared to the use of nanoindentation.

(5) The value obtained using this hardness test method is simply a strength index under a certain set of conditions and is not directly linked to physical theory. However, the success of Rockwell hardness suggests that this is not a major problem in industry.

(6) In the Rockwell hardness test, there is a limit to the hardness range that can be measured depending upon the scale. Unless the scale is changed according to the material, the hardness value for a soft material may sometimes be negative. However, this test method does not have such restrictions.

(7) Depending upon the type of indenter and the ratio r between the testing load and the reference load, free and diversified combinations are conceivable, however because this test method was developed for the purpose of providing industry with a coherent independent hardness scale that does not depend upon the load range, it is necessary to take particular care that many different scales do not appear indiscriminately.

Regarding this test method, it is additionally necessary to carry out studies concerning the effect of differences in the geometry of Vickers and Berkovich indenters, and so on, and also the application of this method to the nanoscope range. However, just as the Rockwell hardness does not contradict the Vickers hardness, this new industrial hardness test proposal does not contradict other industrial hardness test methods, such as nanoindentation, either. Rather, it actively utilizes the merits of nanoindentation, Rockwell hardness, Vickers hardness, and so on, and it goes without saying that it is also intended for use in hardness testing and as an aid to the promotion of research.

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