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TEST-LOCATION SPECIFICATION BY MEANS OF HARDNESS MAPPING ON VICKERS BLOCK SURFACE

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Abstract – The non-uniformity of the hardness reference block is one of the important factors, which influence the hardness measurement. For calibration of hardness reference block, the elementary idea to reduce the error from this factor is to make the indentations at the distributed location as though covering the entire test surface with limited number of indentations.

The principle to decide the appropriate numbers and locations of the measurement is to consider the trend and frequency of hardness distribution. "Stratified sampling" was introduced to the study on the assumption that the confidence of average hardness estimation would be increase with an appropriate test location. Six Vickers hardness reference blocks of 200, 600, 900 HV from 2 different manufacturers were selected for the experiment. The numbers of indentations were made on the entire surface of all blocks with three different levels of the test force. The analysis of hardness distributions was carried out with their measurement data with several aspects of the study.

The possible trends of hardness distribution of the blocks, which, considered in the study i.e., circumferential divisions and radial divisions were selected to view the difference in hardness variation. The effect of stratified conditions to the measurement result was judged by using the analysis of variance (ANOVA).

In most case of the experimental results, both stratifying conditions had significant influences on the reference blocks from both manufacturers with the different trends. Therefore, for higher confidence of hardness number estimation, the idea of test location specification should be taken into account by a considering of both stratifying conditions.

Basically, the minimum numbers of indentations that give the reproducible hardness value upon the repeated measurement is desirable. By varying the stratifying conditions, the observed variations in hardness tended to decrease with the increasing number of strata. From the experiment, more than 6 to 12 strata were recommended for reliable hardness reference block measurement whereas 5 indentations were required as the minimum number in ISO 6507 part 3 [1].

Keywords: Non-uniformity, Stratified sampling, ANOVA

1. INTRODUCTION

The *hardness reference block* is widely used for transfer of hardness standard value, hardness testing machine verification and quality control of measurement accuracy. In these days, the role of reference blocks as the *reference materials* is getting more important because of the requirement for the measurement traceability of the calibration laboratories specified in ISO/IEC 17025 [2]. Therefore adequate study of non-uniformity of reference block is one of the interesting issues. For Vickers hardness, the performance of reference block is especially critical due to wide range of its applicable testing force or indentation depth.

The most of hardness reference blocks are made of steel while copper or aluminium alloys are used for some softer hardness levels and ceramics are used for extremely hard levels. Even if the appearance of reference block is similar, the nature of them may be different because of the manufacturing process. The chemical compound and preparation of raw material will affect the conditions and microstructure of it as the result of the different nature of reference blocks. The heat treatment and surface finish are also critically influencing the characteristics of blocks.

ISO 6507-3 is specifying the procedure of the reference block calibration; on each reference block, five indentations shall be made, uniformly distributed over the test surface. However, this engineering procedure does not seem to be sufficient to get the statistically reliable calibration values. In fact, investigations have been done by several groups of researchers to determine the best way of the reference block calibration and its uncertainty [3 – 5].

In the study, numbers of measurements were made on a reference block to observe the uniformity and the distribution of hardness on its testing surface through the hardness mapping. Those measurement results were divided into 24 groups according to the test locations, which correspond to the stratification of the test surface. The significance of trend of hardness distribution was investigated by means of Analysis of variance (ANOVA) to get the suitable way to locate the measurement positions.

Minimum numbers of indentations that are sufficient for representing the mean hardness value of the reference block were determined from the relation between variations in hardness with respect to numbers of divided sections.

2. INSTRUMENTS AND METHOD

The hardness reference blocks of two different manufactures were prepared for this experiment. The dimensions of those disc-shaped blocks are 64 or 65 mm of diameter and 15 mm of thickness. Akashi Vickers hardness calibration machine model SHT-41 was used to measure the hardness in numbers of locations over the testing surface of those reference blocks. The diagonal lengths and therefore the hardness of each indentation were measured with an automatic measuring system by means of digital image processing. To obtain the suitable magnification on the video screen, the objective lens was chosen from x5 (NA = 0.10), x10 (NA = 0.25), x20 (NA = 0.40) or x40 (NA = 0.65) of magnifications. The measuring method and environmental condition control were in accordance with ISO6507 part 3 including the accuracy of the testing force.

The experiment was carried out on 200, 600 and 900 HV reference blocks with the test force of 9.807 N (1 kgf), 98.07 N (10 kgf) and 294.2 N (30 kgf). Indentations were made in the square area of 46 mm x 46 mm where the motorized translation stage of the hardness machine is available. Due to the limitation of spacing between indentations, the less number of indentations is planned for 200 HV block than other hardness level blocks. Approximately 80 indentations for 200 HV block and 137 indentations for 600 and 900 HV block, were made with each test force. The layout of indentation position of 200, 600 and 900 HV was illustrated in Fig. 1.

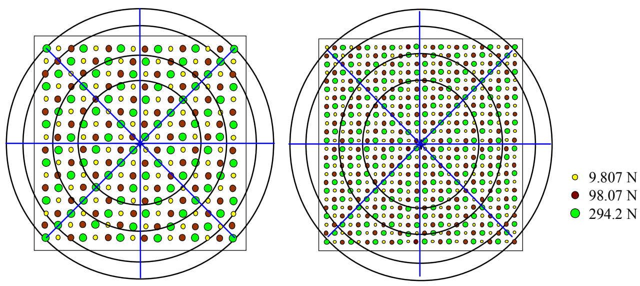

Fig. 1. Layouts of the indentation position

3. RESULTS

3.1. Performance of reference blocks

The averages and standard deviations of all experiments are shown in Table 1. The measurement results could be affected by the error of automatic measurement system and some outliers were obtained. In this summary of Table 1, those abnormalities were already rejected to make the results robust.

The standard deviations range from 0.226 to 2.27 %. For smaller indentations, *e.g.*, 900 HV 1, large amount of scattering was observed. It is known that the repeatability of the hardness machine is getting worse for smaller indentations because of some factors, *e. g.*, smaller size of indentation on the video screen or narrower depth of focus with higher magnification objective lens.

For both manufactures, 200 HV blocks have larger scattering. The hardness of 600 and 900 HV blocks of the manufacturer 2 varied with the testing force. It is showing that the hardness of these blocks was indentation depth dependent and the surface layer of the test surface was possibly harder than the inside of the blocks.

TABLE 1. Summary of reference blocks' performance

Manufacture	Hardness level	Testing force	Number of measurements	Average	Standard deviation
1	200 HV	9.807	80	212.2	2.31
		98.07	76	212.6	3.97
		294.2	82	211.7	3.75
	600 HV	9.807	138	594.3	13.5
		98.07	137	602.9	3.39
		294.2	134	594.2	3.94
	900 HV	9.807	137	864.4	9.04
		98.07	137	878.5	5.39
		294.2	134	877.1	5.13
2	200 HV	9.807	78	190.6	1.27
		98.07	81	196.7	4.15
		294.2	82	193.7	2.44
	600 HV	9.807	137	597.2	8.93
		98.07	137	605.6	2.63
		294.2	134	609.1	1.70
	900 HV	9.807	137	866.1	6.29
		98.07	137	874.1	2.48
		294.2	134	880.8	1.99

3.2. Hardness mapping

The hardness distribution of the reference blocks can be found in contour plots of them. The typical representations of 900 HV reference blocks of two manufacturers are shown in Fig. 2. It is showing that the trend of hardness on the reference block is different for each blocks, *e. g.*, the inside of the testing surface is harder than the outside for the reference block of manufacture 1 since the hardness distribution looks random for the reference block of manufacture 2. If the calibration value of a reference block is assumed to be the average over the whole testing surface defined by

$$\overline{H} = \frac{1}{A} \iint_A H(x, y)dxdy, \qquad (1)$$

where $H(x, y)$ is the hardness at the coordinates of (x, y) and A is the area of testing surface, and the value is estimated with limited number of data, the number and the locations of measurements would affect the estimated average value.

In order to study the suitable way of sampling over the testing surface of reference blocks, each block surface was divided into 24 sections equally in area by 2 concentric circles and 4 diameters providing the map of hardness distribution on the block surface. This technique was expected to be one of the ideas to find out how to locate the test location on the surface of the block in order to obtain the reliable measurement.

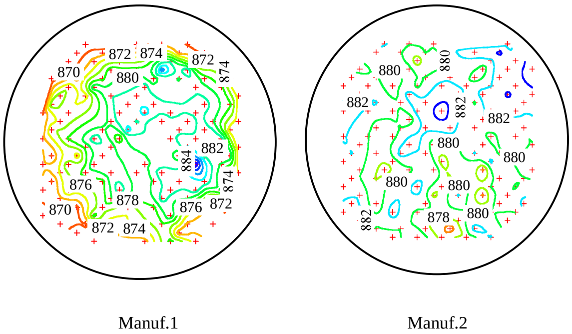

Fig. 2. Examples of the contour plots of 900 HV hardness reference blocks of 2 manufacturers with 294.2 N of testing force.

Fig. 3 shows the average hardness distribution of the stratified areas of the same reference blocks with Fig. 1. The colour level indicated the degree of hardness deviation (in %) from the whole block mean value.

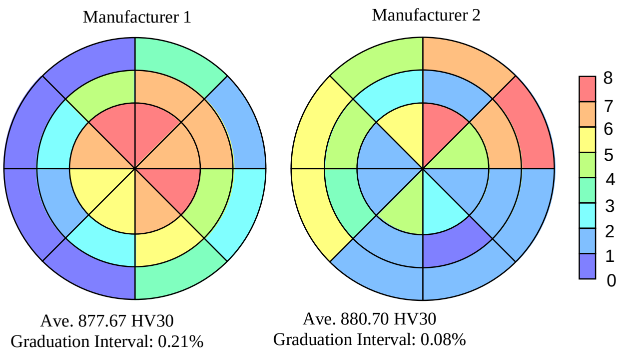

Fig. 3. The maps of hardness distribution of 900 HV hardness reference blocks of 2 manufacturers

If one measurement is made in each area, the calibration value of the reference block is estimated as the arithmetic mean

$$\overline{H} = \frac{1}{n}\sum_{i=1}^{n} H_i \,, \qquad (2)$$

where n is the number of stratified areas, i. e., the number of measurements and H_i is the measured hardness value of each stratum. It is clear that this average of discrete equation will approach to Equation (1), therefore the better estimation of the average value could be obtained, when n is getting larger. The definition of appropriate number and locations of measurements are discussed in the following clauses.

3.3. Significance of non-uniformity

The number and locations of measurements should be chosen with the consideration of the trend and frequency of hardness distribution. In the study, ANOVA (analysis of variance) method was applied to judge the significance of the trend.

To view the difference in hardness variation due to the stratifying conditions, two different stratified condition; circumferential divisions and radial divisions were selected. Each set of data was analysed in the one-way layout and the significance of stratifying conditions were verified with the random effect of experimental error by using ***F***-test.

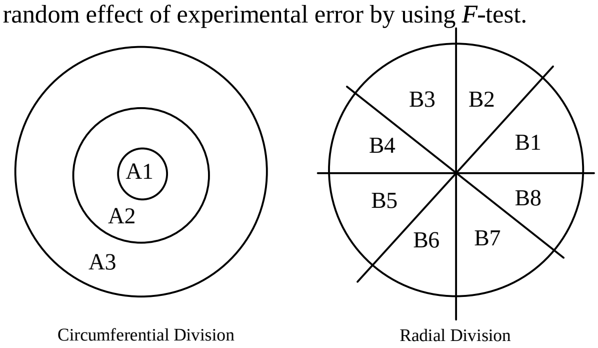

Fig. 4. Stratifying conditions; circumferential and radial division

Ideally, the hardness of test surface should be uniform. The test location should not have the influence on the result of ideal reference hardness block. From the experiment, the characteristic hardness distributions on each block were detected. Figs. 5 and 6 show the difference between the averages of strata divided in radial and circumferential directions, respectively. The bar attached to each value represent the standard deviation of error term in ANOVA at the respective experimental condition, *i. e*, 68 % confidence interval. The results of ***F***-tests are also shown in Table 2. According to the results, the following facts are found. For, 200 HV blocks of both manufacturers, in 4 of 6 conditions, the significant trend was not found for radial divisions because the uniformity was poor. For 600 HV blocks considering the radial divisions, the uniformity was poor with testing force of 9.807 N (1 kgf). The possible reason was that the indentation was small (the uncertainty of the measuring depends on the diagonal length, The smaller indentation, the large uncertainty). For 900 HV blocks, there was a clear trend of radial divisions with Manuf.1; the inner concentric part of block was harder than outer part. However, the uniformity of HV1 was not as good as one of 600 HV.

TABLE 2. Significance of stratifying conditions determined by *F*-test

		Radial division	Circumferential division
Manufacturer 1	200 HV1	*	**
	200 HV10	-	-
	200 HV30	-	-
	600 HV1	-	**
	600 HV10	**	*
	600 HV30	**	-
	900 HV1	*	**
	900 HV10	**	**
	900 HV30	**	*
Manufacturer 2	200 HV1	-	-
	200 HV10	-	-
	200 HV30	*	-
	600 HV1	**	**
	600 HV10	**	*
	600 HV30	**	**
	900 HV1	-	**
	900 HV10	**	**
	900 HV30	**	**

The marks * and ** represent significances at 95 % and 99 % confidence levels, respectively.

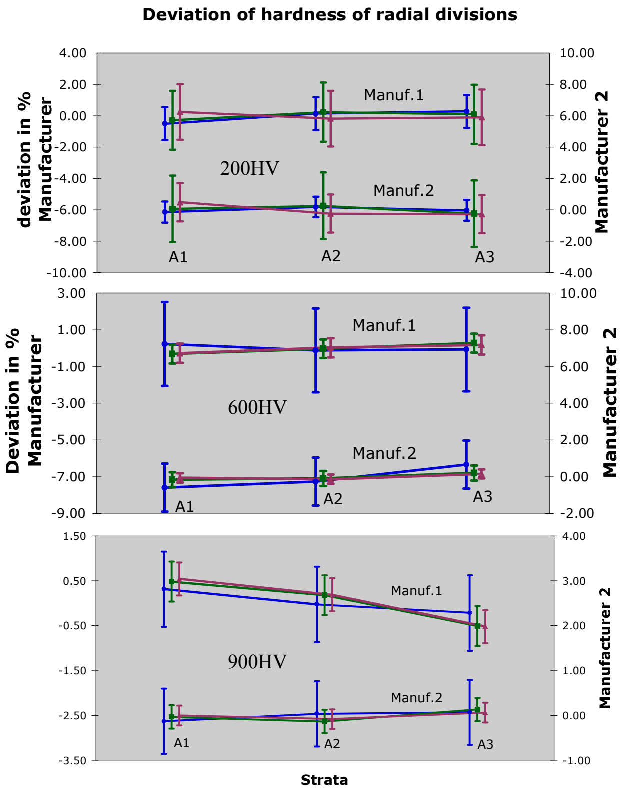

Fig. 5. Hardness deviations due to radial conditions

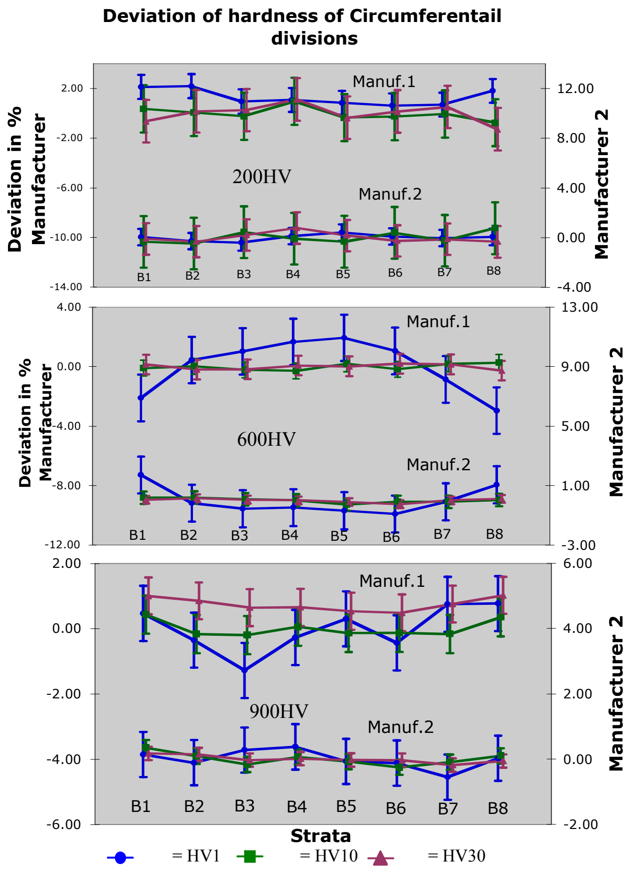

Fig. 6. Hardness deviations due to circumferential conditions

By considering the circumferential divisions, for 200 HV blocks, a significant trend was observed with only one of six curves in the graph (Fig. 6). No trend was detected from this condition. For 600 HV block, both of blocks were detected the significance of stratification on almost all conditions. The trends were observed only for 600 HV1 for both manufacturers. However, this trend was not caused by an experimental error because this trend was not related to the experimental procedure. The significances were obviously detected for all conditions on 900 HV blocks for both manufacturers

3.4. Variation in Hardness with respect to numbers of strata

The purpose of this experiment was to find a best way to sampling hardness value by dividing the test surface of the reference hardness block. The fewest number of indentations made on hardness block surface which leads the reproducible result is wished. In order to satisfy this requirement, the hardness reference block shall be uniform in hardness over an entire surface. Nowadays the fine finished surface of block is not hard to obtained while a whole surface uniformity of block surface is certainly desirable. If the test location can be chosen by "Stratified sampling" as in Fig. 4, the confidence of hardness estimation with fewest indentations will be increased.

The study was carried out under the principle that the hardness trend of reference block could be seen if the sizes of divisions are small enough. In other words, the variation within strata will be minimum if the surface of block is divided into sufficiently small size division.

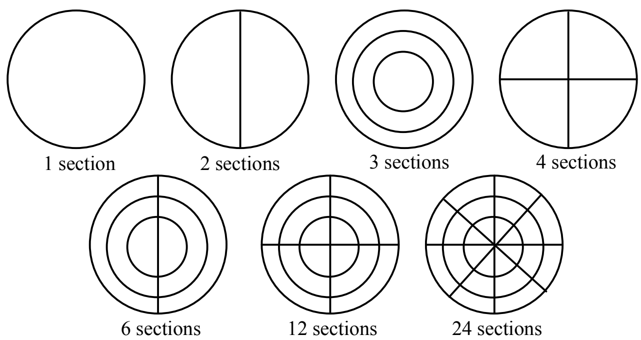

Fig. 6. Stratifying conditions

The study considered the cases where the test surface was divided into 1, 2, 3, 4, 6, 12 and 24 divisions (strata) as all possible subsets of equally divided strata. Fig.6 was plotted to show how the variance in hardness within strata changes due to the number of strata. It could be observed that there were the variations in hardness value according to the numbers of strata. The variation tended to reduce monotonically with increasing number of strata.

For most of the specimens, the variation in hardness has decreased obviously if the block surface was stratified into 6 to 12 strata. From the experiment, more than 6 to 12 strata were recommended for hardness measurement. Up from 12 strata, the variation in hardness was rather stable. Therefore larger number of strata than 12 would not provide the benefit for reducing the hardness variation.

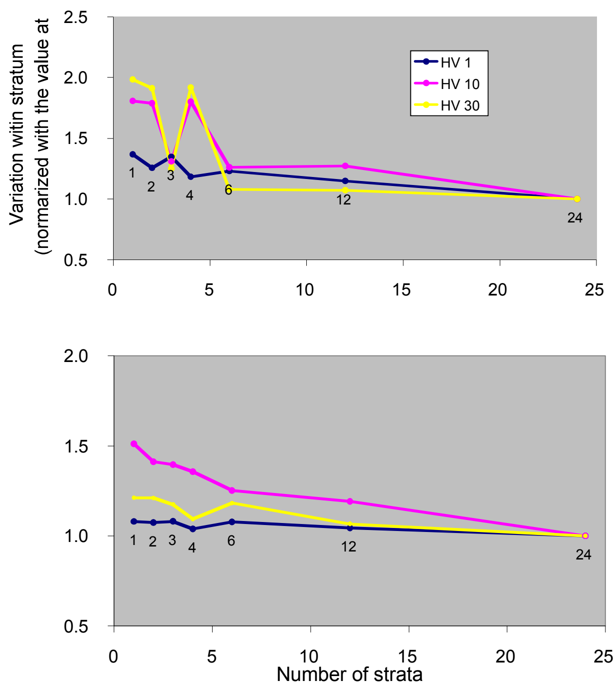

Fig. 7. Variation in HV within the strata

4. DISCUSSIONS

The hardness distribution on a reference block possibly has a trend according to its manufacturing process. The reference block of manufacturer 1 in Fig. 2 is one of the examples. For such reference blocks, the number and locations of measurement on the testing surface are necessary to be chosen carefully. In many cases, one measurement will be made at the center and four indentations at the circumference on the testing surface in order to satisfy the requirement in ISO 6507-3. However, it should be considered that the circumference of the block might be softer than the center and the estimated average hardness of the block could be lower than the true value because more data from soft circumferential areas were used to calculate the arithmetic mean than the data from the center.

As seen in Fig. 7, the variation within stratum is minimized when the testing surface is divided in the radial direction, *i. e*., the stratifying conditions of 3, 6, 12 and 24 divisions.

On the other hand, any apparent trend of hardness distribution was not observed with the reference block of manufacturer 2 in Fig. 2. For such reference blocks, the locations of measurements are not effective to the estimation of average hardness.

However, the stratifying conditions should be concerned whenever the reference block with unknown characteristics is measured. If the test location is not considered to calibrate reference blocks, it should be considered as an uncertainty factor that the estimated average hardness may have some amount of bias.

An average hardness of the block could be changed according to the number of measurement and the test location. Although increasing the number of measurement could reduce the measurement uncertainty, the average of the measurement result may not be able to represent the true hardness value if the test locations were not selected properly. AS a result, suitable sampling gave reliable estimation (unbiased estimation).

5. CONCLUSIONS

The purpose of the study was to find the best way to make measurements on hardness reference blocks and to increase the confidence of the results. The maps of hardness distribution were obtained from measurement data within the grids on the testing surface. In order to investigate the practical way to obtain the number and the locations of measurements, the stratified sampling technique was employed. The significance of difference between stratified divisions, i.e., the trend of hardness distribution, was examined in each of radial and circumferential directions by using ANOVA and *F*-test. The minimum number of measurements was also investigated. Through those considerations, the following results were obtained.

- The standard deviations of all six reference blocks are ranging between from 0.226 % to 2.27 %. The larger amount of standard deviations are observed especially with softer hardness blocks with lower testing force.
- Each hardness block had the different characteristics of hardness variations *i.e*., in radial direction or in circumferential direction. It could be caused by the different manufacturing processes.
- The results of ANOVA show that the variations of hardness are significant in the most cases for 600 and 900 HV blocks whereas they are not significant in the most cases for 200 HV blocks. Both stratifying conditions (radial and circumferential divisions) had significant influence on the reference blocks from both manufacturers, especially higher hardness-level reference blocks. Hence, for reliable measurement, we should consider on both stratified conditions. Consequently, the test locations should be specified by dividing the test block surface in both directions.
- From the experiment of variation in hardness regarded to the number of strata, the minimum number of strata that sufficient to the estimation of hardness number should be more than 6 to 12 and each measurement should be made equally divided area in both radial and circumferential directions.

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