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ON THE LOAD DEPENDENCE OF MICRO-HARDNESS MEASUREMENTS: ANALYSIS OF DATA BY DIFFERENT MODELS AND EVALUATION OF MEASUREMENT ERRORS

The influence of applied loads between 0.09807 N and 0.9807 N on measured values of micro-hardness was evaluated by Meyer's index n, proportional specimen resistance model (PSR) and Hays – Kendall methods, Total Dispersion Zone and Analysis of Variance (ANOVA). The measurement was repeated 6 times using the same hardness reference block with standard hardness H_c = 327 HV0.05 as a sample. The influence of the load on the measured value of micro-hardness is statistically significant, and the relationship between applied load and micro-hardness manifests reverse indentation size effect (ISE) for most of "measurements". The high value of the uncertainty of results can affect the existence and nature of ISE, especially at low loads.

Keywords: micro-hardness, ISE, standard reference block

1. Introduction

Indentation hardness testing is a convenient means of investigating the mechanical properties of a small volume of materials. The principle of Vickers micro-hardness method is identical to (macro)hardness test, except for considerably smaller loads [1].

Like in any test of mechanical properties, there is an obvious requirement for a reliability of measurement results, which is unthinkable without sufficient quality of a measurement process. A perfect measurement would obtain the true value of quantity. True values are, by nature, indeterminable because a perfect measurement cannot be performed. The difference between the true value and the value obtained by a measurement is the error. The uncertainty of the measurement characterizes the dispersion of the values that could reasonably be attributed to the result of measurement. It is inversely proportional to the quality of the measurement [2, 3].

The advantage of Vickers test is the independence of the (macro)hardness on the applied load. However, the micro-hardness of solids depends on the applied load, as a rule. This phenomenon, known as the indentation size effect (ISE) increases the uncertainty of the result of the measurement.

Low load is required when measuring the hardness of small samples, coatings or phases in microstructure [4, 5]. If the low loads are used use of load dependent value of micro-hardness in material characterization results in unreliable conclusions [6].

If a very low load is used, the measured micro-hardness is usually high; with an increase in test load it decreases. Such a phenomenon is referred to as "normal" ISE. It may be caused by the testing equipment [1, 6, 7] or by intrinsic structural factors of the material: work hardening during indentation, load to initiate plastic deformation, elastic resistance and mixed elastic/plastic deformation response of material [1, 5, 7], the effect of indenter/specimen friction resistance, the effect of machining-induced residually stressed measured surface [1, 5 - 8]. In the literature, there are many examples of "normal" ISE occurrence in brittle materials including glass [1].

In contrast to "normal" ISE, a reverse (inverse) ISE (RISE), where the micro-hardness increases with increasing load, is also known. It essentially takes place in materials in which plastic deformation is predominant.

The purpose of this paper is to evaluate the influence of load on the values of micro-hardness measured on the hardness reference block. The existence, nature and size of ISE phenomenon in each measurement were evaluated using Meyer's, PSR, modified PSR and Hays – Kendall methods. The statistical significance of the load on the measured micro-hardness was evaluated by the Analysis of the variance (ANOVA) and Total Dispersion Zone. The measurement was repeated six times (six "measurements") to investigate the variability of obtained results.

2. Experimental material, equipment, and methods

Micro-hardness tester Hanemann, type Mod D32, fitted to microscope Neophot-32 was the equipment. The hardness reference block (or certified reference material CRM) for indirect calibration with specified hardness H_c = 327 HV0.05 and standard uncertainty u_{CRM} = 4.05 HV0.05 was measured sample. The range of applied loads P was between 0.09807 N and 0.9807 N with 0.09807 N step. A researcher performed five indentations (trials) at each load. The result was a "cluster" of 50 indentations in one "measurement", with the minimum distance between indentations according to

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standard [9]. The load duration time was 15 s, the average indentation speed of the indenter in the block was 0.75 mm s^{-1}. The ambient temperature of the laboratory (an also it of the tester and the block) varied between 14.3°C and 21.2°C (TABLE 1). The magnification of the device measuring the dimensions of indentations was 480 ×. The average micro-hardness value of the "measurement" HV, micro-hardness HV0.05, normality (p – value), outliers and relative expanded uncertainty of micro-hardness HV0.05 Urel of particular "measurement" are in TABLE 1. The values of micro-hardness, measured at particular loads are in Fig. 1.

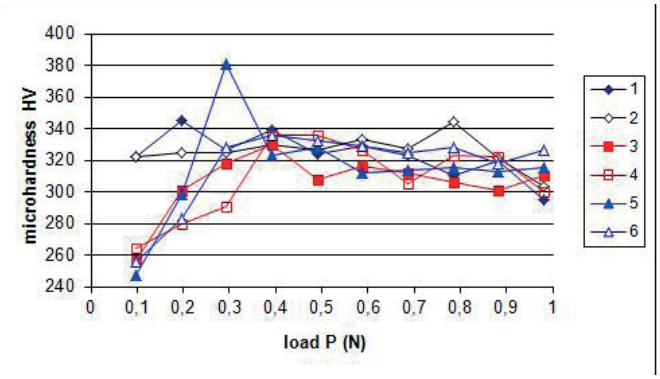

Fig. 1. The relationship between load and micro-hardness

The statistical outliers were detected by Grubbs' test (significance level α = 0.05). Their presence would testify that the process is out of statistical control. The normality was determined by Freeware Process Capability Calculator software (Anderson – Darling test, p ≥ 0.05 for "measurement" with normal distribution). The normality was confirmed only for two "measurements" (No. 2 and 4).

The relative expanded uncertainty Urel of the measured values (Fig. 2) was calculated according to standard [10]. The maximum variability of diagonals and irregularly shaped indentations were much observed at low loads. The ambiguity in the measurement of small indentation areas, particularly with uncertain shape can lead to over- or underestimation of the area of indentation. The result is increased uncertainty just for low loads, which are significant for the evaluation of ISE. The value of the uncertainty may obscure the existence and the character of the ISE phenomenon.

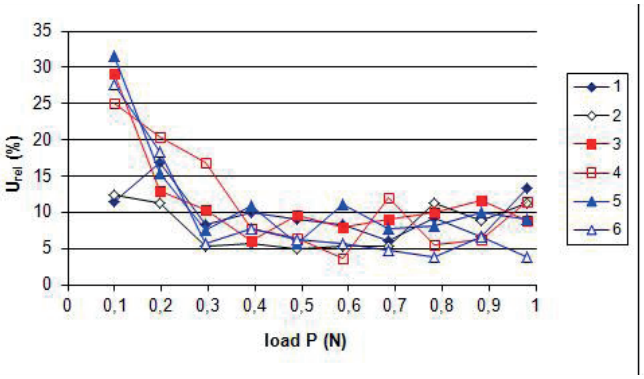

Fig. 2. The relationship between load, sample and relative expanded uncertainty Urel (%)

3. Evaluation of the influence of load on the micro-hardness

3.1. Meyer's power law

The simplest method to describe the ISE phenomenon is Meyer's Law:

$$P = Ad^n \tag{1}$$

The parameters n (Meyer's index or work hardening coefficient) and A are determined by an exponential curve fitting to diagonal d (mm) versus load P (N) in Eq. (1) and are directly connected. From TABLE 2, a continual proportion between A and n can be noted. When n = 2, it gives a value of A = 1897 N mm^{-2}. The index n < 2 for "normal" ISE behavior. When n > 2 there is the reverse ISE behavior and when n = 2, the micro-hardness is independent and is given by Kick's law.

Most of the curves load/micro-hardness in Fig. 1 show increasing of the micro-hardness with applied load to critical values 0.2942 N or 0.3922 N and then remain practically constant. Indentation diagonal length, which corresponds to the critical load, is called the ISE boundary. The exception is "measurements" No. 1 and 2 with an obscure value of the critical load. They are controlled by the Kick's law and, as can be seen in TABLE 2, the value of their index "n" is close to 2. All other "measurements" show reverse ISE phenomenon, typical for plastic materials, as well as metals.

TABLE 1

The ambient temperature, micro-hardness HV - average value of all 50 indentations, micro-hardness HV0.05, outliers, test of normality (p value), relative expanded uncertainty of micro – hardness HV0.05 U_{rel}, the value p and part of the variability explained by the load α of one factor ANOVA

	T (°C)	HV	HV0.05	outliers	normality (p)	U_{rel} (%)	ANOVA (p)	α (%)
1	21.2	323	324	0	0.02463	9.1	0.025466	35.5
2	14.3	326	326	0	0.35084	4.9	0.143488	26.7
3	19.5	306	308	1	0.00001	9.6	1.99E-09	74.7
4	18.1	308	336	0	0.10010	6.4	2.64E-04	85.9
5	17.0	309	328	1	0.00003	5.6	1.07E-07	68.8
6	18.0	316	332	0	0.0	6.1	4.20E-16	88.6

TABLE. 2

The values of indices for Mayer's, PSR and Hays-Kendall methods

Method	Meyer		PSR		Modified PSR			Hays-Kendall	
Index	n	A N mm^{-2}	a_1 N mm^{-1}	a_2 N mm^{-2}	c_0 N	c_1 N mm^{-1}	c_2 N mm^{-2}	W N	A_1 N mm^{-2}
1	1.9371	1315	2.620	1539	-0.1126	19.067	1315	0.0311	1581
2	1.9957	1688	0.910	1664	-0.1191	18.198	1110	0.0178	1656
3	2.1096	2528	-0.981	1685	-0.1066	13.970	1218	0.0018	1628
4	2.1560	3079	-1.998	1763	-0.1819	23.088	985	-0.0009	1659
5	2.1656	3203	-2.191	1774	-0.1137	13.686	1278	-0.1176	1681
6	2.1986	3768	-3.019	1871	-0.1347	15.867	1275	-0.0127	1747

3.2. Proportional specimen resistance model of Li and Bradt (PSR)

The PSR model of Li and Bradt may be considered to be a modified form of the Hays/Kendall approach to the ISE. Several authors [1, 6, 7, 13] have proposed that ISE behavior may be described by Eq. (2):

$$P = a_1 d + a_2 d^2 \tag{2}$$

Li and Bradt pointed out that the parameters a_1 (N mm^{-1}) and a_2 (N mm^{-2}) of Eq. (2) are related to the elastic and plastic properties of the material, respectively [14]. Eq. (2) may be rearranged to the form:

$$\frac{P}{d} = a_1 + a_2 d \tag{3}$$

The parameters a_1, and a_2 of Eq. (3) may be obtained from the plot of P/d (N mm^{-1}) against d (mm). Measured values of a_1 and a_2 are given in TABLE 2. The parameter a_1 characterizes the load dependence of micro-hardness and describes the ISE in the PSR model. It consists of two components: the elastic resistance of the tested material and the friction resistance developed at the indenter facet/tested material interface [1, 7]. The harder material with higher Young's modulus has higher value of a1 [14].

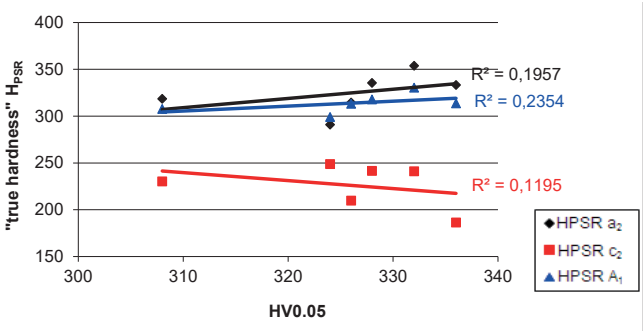

Fig. 3. The relationship between the micro-hardness HV0.05 and "true hardness"

The parameter a_2 is directly related to the load-independent micro-hardness of tested material. It may be a measure of the load-independent "true" micro-hardness which is calculated by multiplying the appropriate parameter by a coefficient 0.1891 [6, 24]. As it can be seen in Fig. 3, the "true hardness" presented using a_2 (H_{PSRa2}) and A_1 (H_{PSRA1}) moderately increases with increasing of micro-hardness HV0.05 with medium correlation. The method of the calculation of the parameter A_1 is presented in chapter 3.4. Calculated "true hardness" is comparable with the values of micro-hardness HV0.05 or average hardness HV.

3.3. Modified PSR

According to energy balance approach parameter c_0 is associated with residual surface stresses in the sample and parameters $c_1 \approx a_1$ and $c_2 \approx a_2$ are related, respectively with the elastic and plastic properties of the tested material [1, 6]. Eq. (4) can be regarded as a modified form of the PSR model.

$$P = c_0 + c_1 d + c_2 d^2 \tag{4}$$

The parameters c_0 (N), c_1 (N mm^{-1}) and c_2 (N mm^{-2}) of (4) may be obtained from the quadratic polynomial regressions of P (N) against d (mm) and their measured values are given in TABLE 2. As it can be seen in Fig. 3, the "true hardness" expressed by c_2 is inversely proportional to micro-hardness HV0.05. A similar tendency was also observed in the case of other materials, e.g. heat-treated steel [20]. It is possible that it is the result of the methodology of the estimation of c_2 by polynomial regression. On the other hand, parameters a_2 and A_1 are determined by linear regression. This fact shows that PSR method is more appropriate for calculation of "true hardness" than modified PSR. The ratio c_1/c_2 is a measure of the residual stresses due to machining and polishing of the tested material. The relationship between c_0 and c_1/c_2 is expected [1]. As can be seen in Fig. 4, the relationship is proportional. It is assumed that the entire surface of the block has been ground and polished, as well. The difference between the residual stress in various locations of the surface of the block characterized by coefficients c_0 or c_1/c_2, therefore, should not be significant. Differences visible in Fig. 4 might be the result of non-uniform chemical composition, microstructure or possibly heat treatment. The inhomogeneity of the sample indicates a variation in micro-hardness in the range between 308 and 336 HV0.05. A ratio c_1/c_2 decreases with decreasing of the micro-hardness. To explain the relationship of the stress will be necessary to measure it using other methods, for example, X-ray diffraction.

To the best of the knowledge of the authors [15], no systematic study on the chemical and physical structure as well as processing of surface (e. g. roughness) of blocks is reported in the literature. It would have a direct bearing on their performance characteristic and hence the uncertainty associated with measured hardness. The investigation made on hardness blocks can lead to a better understanding of their microstructure and can enable to ascertain the critical parameters like the heat treatment/tempering which are utmost important to have single phase material without much segregation and porosity.

Because it is a trade secret, as a rule, it is difficult to obtain information on the chemical composition and methods of heat treatment, machining and polishing of the block. The price and the size of the block limit the possibilities of the analysis.

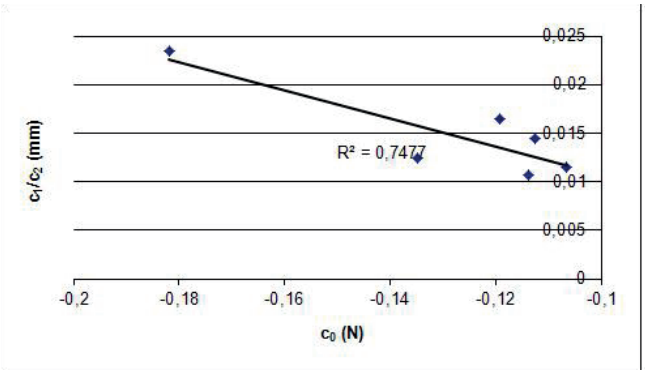

Fig. 4. The relationship between c_0 and c_1/c_2

3.4. Hays – Kendall approach

Hays and Kendall proposed that there exists a minimum test load W (N) necessarily to initiate plastic deformation and below which only elastic deformation occurs. The load dependence of hardness is expressed by Eq. (5)

$$P = W + A_1 d^2 \tag{5}$$

where A_1 (N mm^{-2}) is a constant independent of load. The values of W and A_1 may be obtained from the regressions of P (N) against d^2 (mm) [1] and are given in TABLE 2. The load to initiate plastic deformation (to create visible indentation) varies in the range 0.0 – 0.03 N.

4. Evaluation of the significance of the load on the micro-hardness by ANOVA

According to one-way Analysis of Variance (ANOVA, significance level α = 0.05) with replication the load has statistically significant influence on the measured value of micro-hardness for all "measurements" with the exception of the measurement No 2. The p values and part of the variability explained by the load α are in TABLE 1. Fig. 5 shows a correlation between Meyer's index "n" and log (p). According to two-way ANOVA without replication, the influence of the load on the micro-hardness is statistically significant (p = 0.000137), but the influence of the "measurement" (area of the surface of the tested material) is not (p = 0.091508). A significant p-value took as $p < 0.05$ or $\log(p) < -1.30103$ suggests that the influence of the load on the measured value of the micro-hardness is statistically significant. The statistical significance of applied load and also the index n characterizing the size of ISE increase with decreasing of the value p.

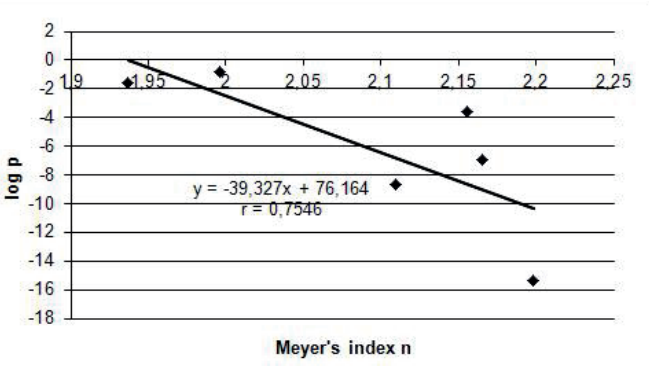

Fig. 5. The relationship between Meyer's index n and logarithm of p-value

5. Total dispersion zone

The value of the Total Dispersion Zone S_M calculated for a particular load evaluates the ability of the "measurement" achieve the same values of the micro-hardness. It is necessary to calculate the average values HV_1, HV_2,…HV_6 and to calculate their standard deviations $s_{\Delta 1}$, $s_{\Delta 2}$ …$s_{\Delta 6}$ for 5 trials in particular "measurement" and particular load [16]. Total scatter zone SM will be calculated by Eq. (6) and by Eq. (9) as a relative value:

$$S_M = \sqrt[6]{\bar{s}^2 + s_v^2} \tag{6}$$

Standard deviation s_v is a standard deviation of 6 average values HV_1, HV_2,..., HV_6. Average standard deviation of all "measurements" at one load will be calculated by Eq. (7) and Eq. (8):

$$\bar{s} = \frac{\bar{s}_\Delta}{\sqrt{2}} \tag{7}$$

$$\bar{s}_\Delta = \frac{s_{\Delta 1}+s_{\Delta 2}+s_{\Delta 3}+s_{\Delta 4}+s_{\Delta 5}+s_{\Delta 6}}{6} \tag{8}$$

$$S_M\% = \frac{S_M}{T} \cdot 100 \tag{9}$$

The sign tolerance T = 65.4 HV in Eq. (9), the same for all test loads, was calculated under maximal permissible error (10 % of 327 HV 0.05) according to standard [10]. We regard SM % as follows: 0 to 20 % good, 21 to 30 % limited usable and more than 30 % unacceptable. As can be seen in Fig. 6, the values of S_M are good with improvement from the load 0.09807 N until the load 0.2942 N or 0.3922 N. The value of S_M% remains practically the same with further increasing of the load. This fact is in good agreement with the results of ANOVA.

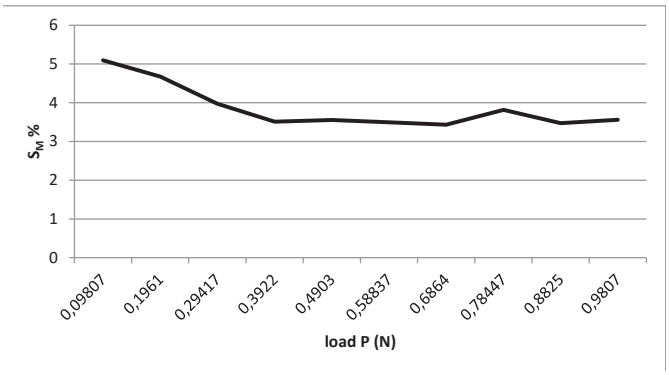

Fig. 6. The relationship between the load and SM

6. Discussion

The temperature is one of the most significant influence quantities in metrology. Vickers test allows calibration in a relatively broad interval of temperatures [10]. Effect of temperature on the measured values of micro-hardness and ISE is ambiguous. Statistically significant effect was observed for blocks with H_c = 195 [11] and 519 HV0.05. Conversely, it has not been confirmed for blocks with H_c = 327 and 392 HV0.05 [12]. The temperature in the laboratory varied between 14.3°C and 26.2°C. The influence of temperature on the mechanical properties of the block is practically negligible in the said range, but the affection of the tester (thermal dilatability) or researcher (personal sense of comfort) are possible. The value of Meyer's index "n" actually increases with increasing temperature. To eliminate temperature as a possible source of variability is appropriate to carry out all measurements at the same temperature (the reference temperature 20°C at best).

Although the block meets the requirements of the standard [17] and, therefore, can be used as a standard reference block, the inhomogeneity of its chemical composition, microstructure, quality of the polishing and residual stresses are possible. They can be a source of observable but not statistically significant variability of the value of Meyer's index "n". The metals (Al, Zn, Cu, Fe, Ni, Co) and reference blocks with standard hardness H_c between 195 HV0.05 and 519 HV0.05 showed reverse ISE at loads between 0.09807 N and 0.9807 N [18, 19, 20].

The used diamond pyramid was viewed under an optical microscope. It is free from surface defects and is without the line of conjunction between opposite faces. Unlike (macro) hardness [21] it is not possible to measure the true value of its vertex. The orientation of the faces of the pyramid is fixed in the micro-hardness tester. Rotation of the diamond as in the case of (macro)hardness tester is impossible [22].

The method of measuring the diagonals may also affect the character of ISE. The measurement of micro-hardness with loads between 0.09807 N and 0.9807 N has been carried out on the reference block with H_c = 242 HV0.05 in direct mode (n = 2.0209, the measuring device was fitted to the micro-hardness tester). The cluster of indentations was thereafter photographed (scanned) for remote mode. Computerized methods ImageJ (n = 2.1151) and TechDig. 1.1.b (n = 1.9846) were used for measuring of diagonals. Finally, the diagonals were measured by slide caliper (scale division 0.01 mm) on the hard copy of indentations (n = 1.8589). The difference between values of micro-hardness obtained by particular modes is statistically significant [23].

7. Conclusions

1. The influence of the load on the measured value of micro-hardness is statistically significant.
2. The relationship between applied load and micro-hardness manifests reverse ISE for most of the measurements.
3. The area of measurement has not statistically significant effect on the presence of ISE.
4. The high value of the uncertainty of the measurement result especially at low loads can affect the existence and nature of ISE.

Acknowledgements

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