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# THE AUTOMATIC TESTERS IN MICROHARDNESS MEASUREMENT AND ISE EFFECT

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**Abstract**: The measurement of micro-hardness with applied loads 0.09807 N, 0.24518 N, 0.49035 N and 0.9807 N has been carried out by five automaticmicrohardness testers. Each appraiser obtained readings of the tester which she/he normally operates.The influence of applied load and equipment on the measured value of micro-hardness and the nature and the size of ISE phenomenon was evaluated by Meyer's index n. The difference between values obtained by particular testers is statistically significant.
**Keywords**: Automatic tester,micro-hardness test, microindentation, CRM

## 1. INTRODUCTION

Measurement of micro-hardness can be carried out in a similar manner to the Vickers macro-indentation tests with diamond pyramid. However, the most important and intractable problemassociated with low loads (the deep of indentation is less than 10 μm as a rule) is that concerned with change in indentation size [1, 2]. The micro-hardness of solids depends on the applied load. The study of relationship between micro-hardness andload has been carried out not only for metallic materials, but also forsemiconductors, glass, slag, ceramics and organic crystals [3-8]. The dependence of measured values of the micro-hardness of solids on the applied load is known as the indentation size effect (ISE). It increases the uncertainty of the measurement result and may result in unreliable conclusions, particularly if low loads (less than 0.294 or 0.392 N as a rule) are used.Low load is required when measuring the hardness of small samples, coatings, thin layers or phases in metallography [9]. When 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 [10, 11] 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 [9, 10, 12], the effect of indenter/specimen friction resistance, the effect of machining-induced residually stressed measured surface [10, 9–13]. In the literature, there are many examples, which reveal that the "normal" ISE occurs in brittle materials including glass [10]. In contrast to "normal" ISE, a reverse (inverse) ISE (RISE), where the apparent micro-hardness increases with increasing load, is also known. The reverse ISE essentially takes place in materials in which plastic deformation is predominant. The purpose of this paper is to evaluate the influence of the load and equipment on the values of micro-hardness, using Meyer's and PSR methods.

## 2. EXPERIMENTAL MATERIALS AND PROCEDURES

Fivetypes of automatic micro-hardness testers (marked as A-E) were used as equipments. The hardness reference block (certified reference material CRM) for indirect calibration with specified hardness $H_c$ = 195 HV0.05 andstandard uncertainty $u_{CRM}$= 4.0 HV0.05 was the sample. The applied loads P were0.09807 N, 0.24518 N, 0.49035 N and 0.9807 N. Each appraiser obtained readings of the tester which she/he normally operates. An appraiser performed five indentations (trials) at each load. The result was the file of 20 indentations. The load duration time was 15 seconds, and the ambient temperature was in accord with the standard [14, 15]. Average values of particular files (HV) are in tab. 1 (HV). The statistical outliers were detected by Grubbs' test (significance level $\alpha$ = 0.05). Their presence would testify that the process is out of statistical control. No outlier was found. Absence ofoutliers suggests that the measurement process has avoided the gross errors.

The normality (determined by Freeware Process Capability Calculator software (Anderson – Darling test, $p \geq 0.05$ for normal distribution) was confirmed for all files, table 1. The values of the micro-hardness obtained at the load 0.49035 N was used for the calibration of the tester in accord with the standard [14].

Average values of micro-hardness, measured at particular loads are in Figure 1 and the values measured by particular tester are in Figure 2.

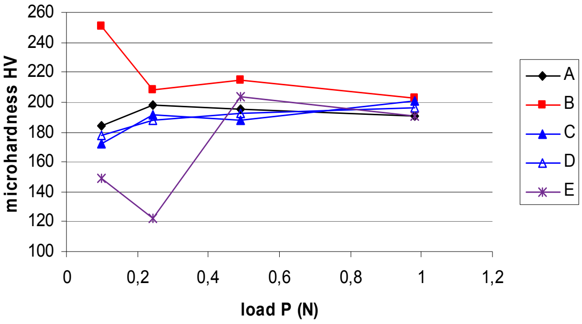

Figure 1. The values of Micro-Hardness

According to the two-factor ANOVA (Analysis of Variance) with replication the equipment (p = $4.68.10^{-24}$) and the load (p = $1.06.10^{-7}$) both have statistically significant effect on the value of measured micro-hardness.

## 3. EVALUATION OF THE INFLUENCE OF THE LOAD ON THE MICRO- HARDNESS

### 3.1 Meyer's Power Law

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

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

The parameters n and $A_{ln}$are determined from a straight line graph of ln d (mm) versus ln P (N). Meyer's index n (work hardening coefficient) are the slope and $A_{ln}$is the y-intercept of the straight line, tab. 2. When n = 2, the micro-hardness is independent of the applied load and is given by Kick's Law. However, n<2 indicated "normal" ISE behavior, and the measured micro-hardness decreases with applied load. When n >2, there is the reverse ISE behavior, measured micro-hardness increases with increasing of the load.

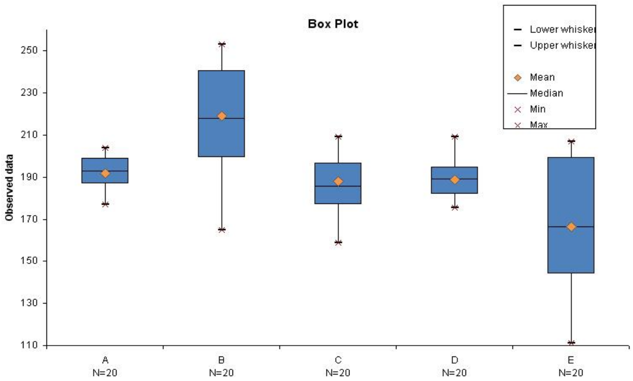

Figure 2. The box-plot, results of particular testers

### 3.2 PSR (Proportional Specimen Resistance model) and modified PSR

Several authors [9, 11] have proposed that ISE behavior may be described by the Eq. (2):

$$P = c_1d + c_2d^2 \tag{2}$$

Gong et al. [9, 11] used an energy balance approach to examine the ISE and rearranged Eq. (2) into modified form of the PSR:

$$P = c_0 + c_1d + c_2d^2 \tag{3}$$

The values of constants $c_0$ (N), $c_1$ (N $mm^{-1}$) and $c_2$ (N $mm^{-2}$)of Eq. (3), obtained from the quadratic polynomial regressions of P/d (N $mm^{-1}$)against d (mm) are given in tab. 2. The parameter $c_1$characterises the load dependence of micro-hardness (elastic properties). Itconsists of the elastic resistance of the test specimen and the friction resistance developed at the indenter facet/specimen interface [16]. The parameter $c_2$ is the measure of the load-independent micro-hardness (plastic properties). The ratio $c_1/c_2$ may be treated approximately as a measure of the residual stress due to machining and polishing [11, 17].

### 3.3 Hays – Kendall approach

Hays and Kendall proposed that there exists a minimum load W(N) necessarilly to initiate plastic deformation and below which only elastic deformation occurs. Then the load dependence of hardness is expressed by Eq. (4), where $A_1$(N mm$^{-2}$) is a constant independent of load.

$$P = W + A_1 d^2 \tag{4}$$

The values of W and $A_1$ may be obtained from the regressions of P (N)against $d^2$ (mm)[10] and their measured values are given in tab. 2. The load to initiate plastic deformation (to create visible indentation) varies in the range 0.014-0.062 N. The constant A1 can be used for calculation of "true hardness"; $H_{PSRA1} = 0.1891\ A_1$.

Table 1. The average micro-hardness value of particular files (HV), the normality (p – value), micro-hardness HV0.05, relative repeatability $r_{rel}$ (%), maximal relative error $E_{rel}$ (%)and relative expanded uncertainty of calibration $U_{rel}$ (%) of the results obtained by particular testers.

| tester | HV | normality | HV0.05 | $r_{rel}$ | $E_{rel}$ | $U_{rel}$ |
|---|---|---|---|---|---|---|
| A | 192 | 0.4297 | 195 | 4.1 | -0.1 | 5.6 |
| B | 219 | 0.5950 | 215 | 1.8 | 10.0 | 14.5 |
| C | 172 | 0.6311 | 188 | 4.7 | -3.5 | 9.9 |
| D | 178 | 0.4107 | 189 | 7.0 | -1.1 | 8.8 |
| E | 176 | 0.0700 | 176 | 2.2 | -4.4 | 9.1 |

Table 2. The values of Meyer's index n and indices $A_{ln}$, $c_0$, $c_1$, $c_2$, W, $A_1$ and "true hardness" $H_{PSRA1}$.

| tester | n | $A_{ln}$ | $c_0$ | $c_1$ | $c_2$ | $c_1/c_2$ | W | $A_1$ | $H_{PSRA1}$ |
|---|---|---|---|---|---|---|---|---|---|
| A | 2.0280 | 7.0326 | -0.041 | 4.91 | 892 | 0.0055 | 0.004 | 1007 | 190 |
| B | 1.8439 | 6.4091 | 0.002 | 2.21 | 999 | 0.0022 | 0.019 | 1054 | 199 |
| C | 2.1275 | 7.4092 | 0.040 | -6.91 | 1241 | -0.0056 | -0.023 | 1076 | 203 |
| D | 2.0888 | 7.2608 | 0.003 | -1.79 | 1095 | -0.0016 | -0.014 | 1052 | 199 |
| E | 2.2596 | 7.7786 | 0.067 | -13.17 | 1365 | -0.0097 | -0.062 | 1064 | 201 |

## 4. TOTAL DISPERSION ZONE

The valueoftheTotal Dispersion Zone $S_M$ calculated for a particular load evaluates the ability of the testers achieve the same values of the micro-hardness. It is necessary to calculate the average values $HV_A$, $HV_B$…$HV_E$andto calculate their standard deviations $s_{\Delta A}$, $s_{\Delta B}$ …$s_{\Delta E}$ of 5 trials of particular tester at particular load [18]. Total scatter zone $S_M$ will be calculated by Eq. (5) and (6) as a relative value:

$$S_M = \sqrt[6]{\bar{s}^2 + s_v^2} \qquad S_M\,\% = \frac{S_M}{T}.100 \tag{5)-(6}$$

Average standard deviation of all values of microhardness under the same load was calculated by Eq. (7) and (8):

$$\bar{s}_\Delta = \frac{s_{\Delta A} + s_{\Delta B} + s_{\Delta C} + s_{\Delta D} + s_{\Delta E}}{5} \tag{7}$$

$$\bar{s} = \frac{\bar{s}_\Delta}{\sqrt{2}} \tag{8}$$

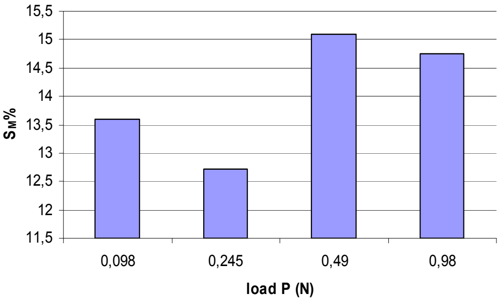

Figure 3. The Values $S_M$%

$s_v$ is a standard deviation of 5 average values $HV_A$, $HV_B$..., $HV_E$ measured under the same load.

The sign tolerance T = 39 HV in Eq. (6), the same for all test loads,was calculated pursuant to maximal permissible error (10 % of 195 HV 0.05) according to standard[14]. We regard $S_M$ % as follows: 0 to 20 % good, 21 to 30 % limited usable and more than 30 % unacceptable. As can be seen in Fig. 3, the values of $S_M$ % are "good" for all four applied loads. The differences between the results of hardness obtained by particular tester are not significant under this method. This fact is not in good accord with the results of ANOVA. It seems that the method of Total Dispersion Zone is not sensitive enough for this type of measurement.

## 5. DISCUSION

The influence of the appraiser on the result is marginal with respect to automatic function of testers. High variability of the measured values was observed despite the same sample and automatic measurement system. The sample was standard hardness block with expected high homogeneity of micro-hardness and with uniform residual stress due to machining and polishing of its surface. Both equipments and test loads have statistically significant effect on the micro-hardness. The result is that the same sample shows simultaneously “normal” and reverse ISE. In the literature, there are many examples, which reveal that the “normal” ISE occurs in brittle materials as non-metals, semiconductors or glass. Just “normal” ISE was detected by the tester with a maximal error $E_{rel}$ and maximal uncertainty $U_{rel}$ (the tester B).

As it was used the same sample, the ISE obviously may be caused by the testing equipment. The experimental errors resulting from the measurement of indentation diagonals as a result of the limitations of the resolution of the objective lens, inadequate measurement capability of small areas of indentations anddetermination of the applied load are typical causes of “normal” ISE [10, 11, 12]. As for the tester, reverse ISE can be explained by effects of vibration and indenter bluntness at low loads [9].Vickers method allows calibration in relatively broad interval of temperatures (23°C ± 5°C) [14]. In this interval, the temperature was found to influence the value of the index n [19]. A variability of the nature (“normal” and reverse) of ISE were observed on the same block measured manually by Hanemann tester. The value of n varied between 1.874 and 2.360 [19]. High variability of n was observed also in repeated measurements of more hard (up to 392 HV0.05) reference blocks, for example [20].

## 6. CONCLUSIONS

The variability of measured values of the micro-hardness values and parameters ISE is high despite the use of automatic hardness testers with practically excluding the impact of the researcher. The influence of the type of the tester is statistically significant. The results of testers with high uncertainty show extreme values of Meyer’s index.

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