THE AUTOMATIC TESTERS IN MICROHARDNESS MEASUREMENT AND ISE EFFECT

Jozef Petrík[1)], Peter Blaško[1)], Jana Bidulská[1)], Anna Guzanová[2)], Iveta Sinaiová[3)]*
[1)]Technical University of Košice, Faculty of Metallurgy, Košice, Slovakia
[2)]Technical University of Košice, Faculty of Mechanical Engineering, Košice, Slovakia
[3)] Institute of Material Research, Slovak Academy of Sciences, Košice, Slovakia

Received: 11.07.2016
Accepted: 16.09.2016

[*]Corresponding author: *e-mail: jozef.petrik@tuke.sk Tel.: +421 55 602 2872, Institute of Materials, Faculty of Metallurgy, Technical University of Košice, Letná 9, 042 00 Košice, Slovakia*

Abstract
The measurement of micro-hardness with the applied loads 0.09807 N, 0.24518 N, 0.49035 N and 0.9807 N has been carried out by three automatic and one manually-operated micro-hardness testers. The certified reference material (CRM) was the tested sample. Each operator obtained the readings of the tester, which she/he normally operates. The measurement was repeated after thirty months. The influence of the testers and their stability, as well as applied load on the measured values of the micro-hardness and the indentation size phenomenon (ISE), were evaluated. The parametric and non-parametric tests, Analysis of Variance (ANOVA), Z-score and Total Dispersion Zone were used for the evaluation of the statistical significance of obtained factors. The ISE was evaluated using Meyer's and Proportional Specimen Resistance model and also by Hays – Kendall approach. The variability of measured values of the micro-hardness and parameters of ISE is high despite the use of automatic hardness testers with practically excluding the impact of the operator. The results are affected by operators, used testers and by applied loads. The measurement system can't be considered to be stable.

Keywords: hardness, ISE, CRM, iron, repeated measurement

1 Introduction

Measurement of micro-hardness can be carried out in a similar manner to the Vickers macro-indentation tests with the diamond pyramid. However, the most important and intractable problem associated with low loads (the deep of indentation is less than 10 μm as a rule) is that concerned with a change in indentation size [1, 2]. The micro-hardness of solids depends on the applied load. The study of the relationship between micro-hardness and load has been carried out not only for metallic materials but also for semiconductors, glass, slag, ceramics, sintered materials 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 micro-hardness and may result in unreliable conclusions, particularly at low loads. They are required for the purpose of the measuring of small samples, coatings, thin layers or phases in metallography [9].

The measured value of the micro-hardness is usually high if a low load is used; it decreases with an increase in applied load. Such phenomenon is called as "normal" ISE. It may be caused by

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

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 residual stress as the effect of the machining [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 repeatability is the condition of measurement, out of a set of conditions that includes the same measurement procedure, same operators, same measuring system, same operating conditions and same location, and replicate measurements on the same or similar objects over a short period of time [14].

An important aspect of maintaining the capability of the equipment to produce traceable and reliable measurement results is a determination of the maximum period that should be permitted between successive calibrations [15] - the interval of the calibration. Its length is related to the stability of the tester.

The stability (or drift) is the total variation in the measurements obtained with a measurement system on the same standard or parts when measuring a single characteristic over an extended period. The measurement of stability is the change in bias over time. Knowledge of the equipment and measurement conditions help identify special causes when the system is unstable. Stability of measuring equipment is its property, whereby its metrological properties remain constant in time. It may be quantified by the duration of a time interval in which a metrological property or a quality is changed in a defined range. Stability is the absence of special causes of variation; the property of being in statistical control. It refers to both statistical stability of measurement process and measurement stability over time. Both are vital for a measurement system to be adequate for its intended purpose. Statistical stability implies a predictable, underlying measurement process operating within common cause variation (in-control). Measurement stability addresses the necessary conformance to the measurement standard or reference over the operating life (time) of the measurement system [14, 16].

The validity of the three assumptions is analyzed in the present article:

1. The tester has no statistically significant effect on the value of the measured micro-hardness.
2. The applied test load, in the relation to the Kick's law, has no statistically significant effect on the measured value of the micro-hardness. Otherwise, the presumption of the validity of Meyer's law is accepted.
3. The differences between the values of the micro-hardness, repeatedly measured over a long period (the period between trial No. 1 and trial No. 2 was 30 months), under the same conditions are not statistically significant. In this case, the measurement system can be considered stable.

To confirm or refute these assumptions were used the following statistical methods: Parametric and Non-parametric tests, Analysis of Variance (ANOVA), Z-score and Total Dispersion Zone. Existence, nature and size of the above mentioned ISE (statistically significant effect of the load on the micro-hardness) were evaluated by Meyer's, Proportional Specimen Resistance Model, and also by Hays – Kendall approach.

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

2 Experimental material and methods

The experiment consists of two trials; Trial No. 1 and Trial No. 2. There was thirty months period between each trial. Three types of automatic micro-hardness testers operated by one operator, respectively (Duramin marked as D1/D2, Shimadzu marked as S1/S2, and Leco marked as L1/L2) and manually-operated tester (Hanemann; type Mod D32 fitted to microscope Neophot-32, served by two operators and marked as HB1/HB2 and HP1/HP2) was used as the measurement equipment. The index 1 and 2 of tester's marks characterize the number of trials. The hardness reference block (certified reference material CRM) for indirect calibration with specified hardness H_c = 195 HV0.05 and standard uncertainty u_{CRM} = 4.0 HV0.05 was the tested sample. It is difficult to obtain the information about the chemical composition and the methods of heat treatment, machining and polishing of the CRM due to the trade secret. The sample dimension and the effort to minimal intervention in the sample limits the possibilities of chemical and microstructural analysis. The basic material is the iron (ferrite) doped with 0.12 Ni, 0.49 Mn, 0.11 Cr and 0.47 Si. The chemical composition was analyzed by spectroscope Niton XL3 Goldd with validation by SEM MIRA Tescan – EDS OXFORD. The restricted area of the mirror smooth surface of the CRM was etched by Nital. The mean diameter of the grains is 0.049 mm, ranged between 0.022 and 0.081 mm.

The characteristic of trial No. 1 is following: The applied loads P were 0.09807 N, 0.24518 N, 0.49035 N and 0.9807 N. Each operator obtained readings of the tester, which she/he usually operates. An operator performed five indentations at each load. The result was the file of 20 indentations. The load duration time was 15 seconds, and the ambient temperature and the loading rate of automatic testers were in agreement with the standards [17, 18]. Some data concerning the trial No. 1 were presented in the paper [19].

To determine the stability of the equipment, the trial was repeated thirty months after trial No. 1 and was named as trial No. 2. The trial No. 2 was carried out by the same equipment, by the same operators on the same sample, and by the same conditions.

Table 1 The tester Hanemann: loading rate v_1 and the speed of the penetration of the indenter v_2.

	v_1 (N s^{-1})	**v_2 (μm s^{-1})**
HP1	0.1176	0.9035
HP2	0.1320	0.9897
HB1	0.1477	1.1763
HB2	0.1514	1.2783

The penetration speed was calculated only for manually-operated Hanemann tester. The penetration speed of the indenter into the test piece v_2 was calculated by dividing the depth of indentation (1/7 of the mean length of the indentation diagonals) with the time elapsed between the initial application of the load and the full test load. As shown in **Tab. 1** the loading rate v_1 (N s^{-1}) and the speed of the penetration of the indenter v_2 (μm s^{-1}) of both operators are different and moreover change with time.

Mean values of the micro-hardness of individual files (HV) and their standard deviation (s) are in Tab. 2 (HV). The statistical outliers were detected by Grubbs' test (significance level α = 0.05). No outlier was found. The absence of outliers suggests that the measurement process has avoided the gross errors and is under statistical control.

The normality (determined by Freeware Process Capability Calculator software (Anderson – Darling test, if $p \geq 0.05$ the distribution is normal) was confirmed for all files, Tab. 2. All files

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

have a normal distribution. But the set of all measured values (n = 200) has other than the normal distribution.

Table 2 Mean values of the micro-hardness of individual files (HV), their standard deviation (s), P_1 – the test of the normality: p-value, the repeatability r_{rel}, maximum error of the tester E_{rel}, relative expanded uncertainty of the calibration U_{rel}, and p_2 as the p-value indicating the statistical significance of the load by ANOVA

	HV	**S (HV)**	P_1	$\mathbf{r_{rel}}$ **(%)**	$\mathbf{E_{rel}}$ **(%)**	$\mathbf{U_{rel}}$ **(%)**	$\mathbf{p_2}$
D1	172.08	21.49	0.222288	7.7	-7.3	14.9	5.96E-07
D2	202.75	24.26	0.189832	14.18	0.29	13.32	0.29635
S1	230.7	21.88	0.386124	13.93	16.82	30.74	0.000105
S2	229.7	25.2	0.398284	4.7	1.58	17.14	2.18E-06
L1	188.84	9.73	0.396606	7.04	-1.12	8.76	0.005441
L2	196.93	4.15	0.363588	0.64	0.71	5.15	1.85E-02
HP1	189.36	13.93	0.200147	3.92	-4.84	10.27	5.97E-01
HP2	197.63	15.86	0.405229	8.1	5.63	14.97	0.073715
HB1	186.85	23.54	0.546476	8.7	2.32	11.63	2.18E-06
HB2	188.21	23.29	0.523330	5.51	-7.8	13.97	1.37E-07
Trial 1	193.57	27.04	0.077530	-	-	-	-
Trial 2	203.05	24.33	0.028140	-	-	-	-
All results	198.31	26.09	0.008841	-	-	-	-

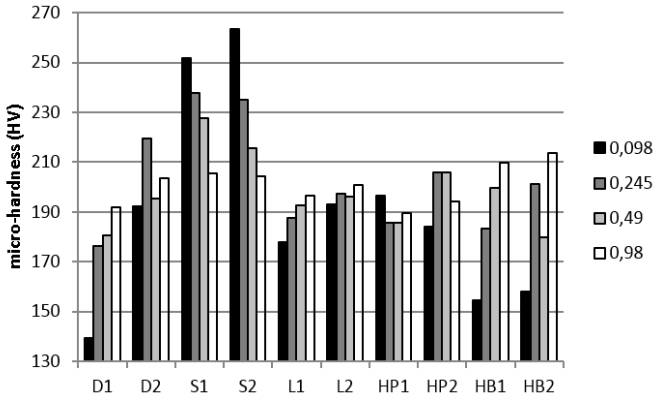

Fig. 1 Mean values of the micro-hardness, measured in both trials

The values obtained at the load 0.49035 N was used for the calibration of the tester in conformity with the standard [17]. The results of the calibration are shown in **Tab. 2**. According to the standard, the results of the calibration shall not exceed 9 % for the repeatability r_{rel}, ± 10 % for the maximum error of the tester E_{rel}, 10 % for the relative expanded uncertainty of the calibration U_{rel}. These requirements of the standard met only for equipment L in both trials. The uncertainty of the calibration decreased in the trial No. 2 for all automatic testers. Manually-

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

operated testers have the opposite tendency, probably as a consequence of the most significant influence of the operator. Mean values of micro-hardness, measured at individual loads by different testers in both trials, are in **Fig. 1**.

The statistical significance of the applied load on the micro-hardness was tested by one way Analysis of Variance (ANOVA). Namely, if the value of p (p_2 in Tab. 2) is less than 0.05, the influence is significant.

3 Results

3.1 Evaluation of the influence of the load on the micro-hardness

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) is calculated using the slope of the line. The value of A_{ln} is the y-intercept of the straight line, **Tab. 3**. If 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. If n >2, there is the reverse ISE behavior.

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

$$P = a_1 d + a_2 d^2 \quad (2.)$$

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 [6]. Eq. (2) may be rearranged in the form:

$$P/d = a_1 + a_2 d \quad (3.)$$

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 **Tab. 3**.

Table 3 The values of Meyer's index n and indices A_{ln}, a_1 and a_2

	n	**A_{ln}**	**a_1**	**a_2**	**a_1/a_2**
D1	2.2983	7.9863	-4.3804	1156.3	-0.00379
D2	2.0141	7.0255	0.0934	1060.1	8.81E-05
S1	1.8414	6.4498	3.5340	983.81	0.003592
S2	1.7990	6.2708	3.8153	955.23	0.003994
L1	2.0888	7.2608	-1.4884	1086.9	-0.00137
L2	2.0297	7.0676	-0.5845	1076.6	-0.00054
HP1	1.9741	6.8018	0.0969	989.8	9.79E-05
HP2	2.0507	7.1521	0.0486	1044.8	4.65E-05
HB1	2.3065	8.1136	-5.0050	1279.4	-0.00391
HB2	2.2248	7.7952	-3.9599	1225.9	-0.00323

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

$$P = c_0 + c_1 d + c_2 d^2 \quad (4.)$$

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

The values of constants c_0 (N), $c_1 \approx a_1$ (N mm^{-1}) and $c_2 \approx a_2$ (N mm^{-2}) of Eq. (4), obtained from the quadratic polynomial regressions of P/d (N mm^{-1}) against d (mm) are given in **Tab. 4**. The parameter c_1 characterizes the load dependence of micro-hardness (elastic properties). It consists of the elastic resistance of the test specimen, and the friction resistance developed at the indenter facet/specimen interface [6]. The parameter c_2 is the measure of the load-independent micro-hardness (plastic properties). The ratio a_1/a_2 or c_1/c_2 may be treated approximately as a measure of the residual stress due to machining and polishing [11, 20].

Table 4 The values of c_0, c_1, c_2, W, A_1 and "true hardness" H_{PSRA1}.

	c_0	c_1	c_2	c_1/c_2	W	A_1	H_{PSRA1}
D1	-0.0172	-2.3996	1107.0	-0.00217	-0.0404	1052	199
D2	0.0245	-2.7383	1129.8	-0.00242	0.0004	1063	201
S1	-0.0548	10.8290	786.4	0.01377	0.0360	1056	200
S2	-0.0062	4.6846	931.0	0.00503	0.0327	1048	198
L1	0.0026	-1.7924	1094.5	-0.00164	-0.0138	1052	199
L2	0.0158	-2.5070	1125.9	-0.00223	-0.0065	1066	201
HP1	0.0323	-3.8492	1090.4	-0.00353	-0.0026	1000	189
HP2	-0.0913	10.9990	767.4	0.01433	0.0087	1027	194
HB1	-0.0108	-3.7486	1247.5	-0.00300	-0.0455	1158	219
HB2	0.1841	-25.3700	1767.4	-0.01435	-0.0441	1149	217

Hays and Kendall proposed that there exists a minimum load W (N) necessary to initiate plastic deformation and below which only elastic deformation occurs. Then the load dependence of hardness is expressed by Eq. (5), where A_1 (N mm^{-2}) is a constant independent of load.

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

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. 4**. The constant A_1 (and also a_2 and, if necessary not very reliable c_2) can be used for calculation of "true hardness" $H_{PSRA1} = 0.1891\ A_1$. The load independent "true hardness" is free of the ISE effect. It is corresponding to the hardness obtained for an "infinite" load and characteristic length $d^* = a_1/a_2$ (or $d^* = c_1/c_2$) [21].
Two-way ANOVA without replication was used for evaluation of the statistical significance of the effect of the tester and the trials on Meyer's index n. The effect of the tester is statistically significant (p = 0.0481) and the trials are not statistically significant (p = 0.25134).

3.2 Total Dispersion Zone

The value of the Total Dispersion Zone S_M evaluates the ability of the testers achieve the same values of the micro-hardness for an individual load. It is necessary to calculate the mean values HV_D, HV_S, HV_L, HV_{HP}, HV_{HB} and to calculate their standard deviations $s_{\Delta D}$, $s_{\Delta S}$, $s_{\Delta L}$, $s_{\Delta HP}$, $s_{\Delta HB}$ of five repeated measurements of the individual tester at different load [22].
Total scatter zone S_M will be calculated by Eq. (6) and (7) as a relative value:

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

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

$$S_M\% = \frac{S_M}{T} \cdot 100\% \quad (7.)$$

Mean standard deviation of all values of micro-hardness under the same load was calculated by Eq. (8) and (9):

$$\bar{s}_\Delta = \frac{s_{\Delta D}+s_{\Delta S}+s_{\Delta L}+s_{\Delta HP}+s_{\Delta HB}}{5} \quad (8.)$$

$$\bar{s} = \frac{\bar{s}_\Delta}{\sqrt{n}} \quad (9.)$$

s_v is a standard deviation of 5 mean values HV_D, HV_S..., HV_{HB} measured under the same load. The sign tolerance T = 39 HV in Eq. (7), the same for all test loads, was calculated under maximal permissible error (10 % of 195 HV 0.05) according to standard [17]. 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. 2**, the values of S_M % are "good" for all four applied loads and both trials. The differences between the results of hardness obtained by the individual tester are not significant by this method.

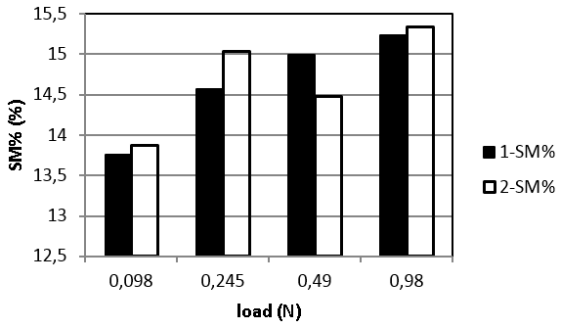

Fig. 2 Total Dispersion Zone - the values of S_M%

3.3 Z-score

The graphical method Z-score, employed for the visualization of results is routinely applied in inter-laboratory comparisons (round-robin tests).

$$z_i = \frac{x_i - \bar{x}}{s} \quad (10.)$$

$\underline{x}_i$ is the mean micro-hardness, measured at individual load by an individual tester in one trial, x is specified hardness H_c = 195 HV0.05 and „s" is the standard deviation of all (n = 200) measured values, **Tab. 2**. The results $|z_i| \leq 2$ are satisfactory and $|z_i| \geq 3$ are unsatisfactory [23, 24]. As can be seen in **Fig. 3**, unsatisfactory results were not observed. The results are better in the trial No. 2. The increasing of the load also improves the results.

3.4 Analysis of Variance (ANOVA), parametric and non-parametric hypothesis tests

Files of values measured by individual testers in the trials No. 1 and 2 have normal distributions. But the file containing all values (n = 200) has other, very likely 2-Parameter Logistic

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

distribution. The same distribution also has the file of the results, measured in the trial No. 2 (**Tab. 2**). If the input data are non-normally distributed, it is appropriate to use non-parametric (robust) tests. Hence, if parametric t-test or the Analysis of Variance (ANOVA) is used, the results are not reliable.

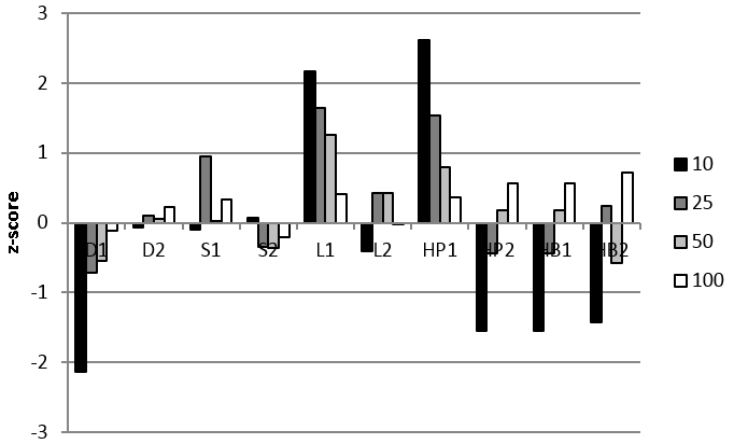

Fig. 3 Z-score

The t-test assesses whether the means of two groups are statistically different from each other. This analysis is appropriate whenever you want to compare the means of two groups. A paired t-test is used to compare two population means where you have two samples in which observations in one sample can be paired with observations in the other specimen (measured at the same load).

The Mann-Whitney and Kruskal-Wallis tests are used to verify the hypothesis that two or more samples were drawn from the same distribution. The Mann-Whitney test is used for two samples. The Kruskal-Wallis test is used when there are two or more samples. Thus, these non-parametric tests are commonly used to determine whether medians, not means, are different between comparison files. For both tests, the statistic test only depends on the ranks of the observations in the combined sample, and no assumption about the distribution of the populations is made. It is the meaning of the non-parametric term in this context.

Median test (or Westenberg-Mood's test) compares the medians of two or more samples. It is "crude" alternative of Kruskal-Wallis test with worse power. It is very robust against outliers, and fairly robust against differences in the shapes of the distributions [25].

Using all above mentioned test (paired t-test, Mood's median test, Kruskal-Wallis test and Mann-Whitney test) the difference between the values obtained in trial No. 1 and No. 2 are statistically significant.

The results of hypothesis tests for individual trials are in **Tab. 5**. The difference between files is not statistically significant according to paired t-test (1) and Mann-Whitney test (2). Other differences are significant. According to Kruskal-Wallis test, it can be greater than 99.99 % confident that Medians are different for both trials. As far as Mood's median test, there are not enough observations that are greater than the median for trial No. 1. Therefore, the test could not be used. For the trial No. 2, all differences are statistically significant according to this test.

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

Table 5 The results hypothesis tests – the difference between files is not statistically significant according to paired t-test (1) and Mann-Whitney test (2)

	Trial No. 1				**Trial No. 2**			
	D	**S**	**L**	**HP**	**D**	**S**	**L**	**HP**
HB	2	*	1, 2	1,2	2	*	1,2	1,2
HP	*	*	1, 2	*	1,2	*	1,2	*
L	*	*	*	*	1,2	*	*	*
S	*	*	*	*	*	*	*	*
D	*	*	*	*	*	*	*	*

Two way ANOVA with replication was used for evaluation of the statistical significance of the load, tester and the trial on the measured value of the micro-hardness. In the trial No. 1 the tester ($p = 4.63\ E^{-20}$) and also the load ($p = 5.30\ E10^{-5}$) have statistically significant influence on the micro-hardness. The tester has a significant effect ($p = 1.55\ E^{-7}$) in the trial No. 2, but the effect of the load is not significant ($p = 0.70012$) on the micro-hardness. The interaction between the tester and the load is statistically significant in the trial No. 1. Taking into account all measured values, both the trial ($p = 0.00074$) and the tester ($p = 1.80\ E^{-21}$) have statistically significant effect on the micro-hardness.

4 Discussion

The assumption No. 1 (about the tester) can be rejected. The tester has a significant effect on measured value of the micro-hardness. It seems that the Total Dispersion Zone is not a sufficiently sensitive method for the measurement of the micro-hardness. The effect of the tester may be connected to different and, in general, high level of their uncertainty. This observation supports the fact that 4/5 of the used testers, including automatic, do not meet the requirements of the standard. The uncertainty of micro-hardness affects the evaluation of the ISE; therefore it can be the basis for decisions relating to the assumption No. 2. Petrík and Palfy in [26] investigated the relationship between the ISE and the uncertainty of the micro-hardness tester.

The assumption No. 2 (about the negligibility of the load) can also be rejected. The load has statistically significant effect on the measured value. This finding corresponds to the conclusion presented almost in all works cited in this paper. Detected ISE is the result of this impact. The problem is its nature and size. The sample with defined properties (CRM!) was used, however, normal and reverse ISE were detected. The tester influences the effect of the load on the micro-hardness. The evidence of this fact is a statistically significant amount of interaction between the load and the hardness in the trial No. 1. The Meyer's index is close to 2 for testers L and HP and the load affect the micro-hardness only negligibly. On the contrary, the results of tester S show a significant normal ISE while the results of the tester HB show significant reverse ISE. The influence of the operator is more significant than the impact of the equipment when using the manually-operated tester H.

The assumption No. 3 (the stability of testers) is also rejected. The changes of the mechanical properties of the sample over time are unlikely. This fact is the result of the progressive decrement of the metrological characteristics of the testers. Therefore the measurement system cannot be considered to be stable.

The influence of the operator on the result is marginal for the automatic function of testers. High variability of the measured values was observed despite the same sample and automatic

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

measurement system. The sample was the CRM with high uniformity of the microstructure and chemical composition. Therefore, it is expected that its micro-hardness and the residual stress due to machining and polishing of its surface are also uniform.
For that reason, the testing equipment apparently may cause the ISE. The experimental error resulting from the measurement of the indentation diagonals is due to the limitations of the objective lens resolution, inadequate measurement capability of small areas of indentations and the determination of the applied load. They can also affect the nature and the size of the ISE [10, 11, 12].
A variability of nature (normal and reverse) of ISE was observed on the same block and was measured manually by Hanemann tester. The value of n varied between 1.874 and 2.360 [26]. High variability of n was also observed in repeated measurements of more hard (up to 392 HV0.05) reference blocks, for example [27].

5 Conclusion

All three assumptions mentioned in the introduction can be rejected. The variability of the micro-hardness and ISE parameters are affected by applied load and by hardness tester. 4/5 of the used testers, including automatic, do not meet the requirements of the standard. Despite the using of automatic hardness testers with practically excluding the impact of the researcher, this variability was not solved. Moreover, these parameters vary with the time. The differences between the values of the micro-hardness repeatedly measured over a long period under the same conditions show that the measurement system is not considered as stable. Therefore, the repeated calibration of the tester with the calculation of the uncertainty and the ISE, as well as, the determination of the appropriate calibration interval due to the expected drift are necessary for the control of the measurement process.

References

[1] X. Cai, X. Yang, P. Zhou: Journal of Materials Science Letters, Vol. 16, 1997, p. 741-742
[2] P. M. Sargent: *Use of Indentation Size Effect on Microhardness for Material Characterization*, first ed., ASTM, Philadelphia, 1985
[3] G. Golan et al: Journal of Optoelectronics and Advanced Materials, Vol. 2, 2000, No. 4, p. 317-325
[4] S. Layar, P. Selvarajan: Recent Research in Science and Technology, Vol. 2, 2010, No. 10, p. 77-81
[5] A. R. Kumar, S. Kalainathan: Journal of Physics and Chemistry of Solids, Vol. 71, 2010, p. 1411-1415, doi: 10.1016/j.jpcs.2010.06.009
[6] H. Kim, T. Kim: Journal of the European Ceramic Society, Vol. 22, 2002, p. 1437-1445, doi: 10.1016/S0955-2219(01)00457-5
[7] T. Kvačkaj, J. Bidulská, R. Kočiško, R. Bidulský: Effect of Severe Plastic Deformation on the Properties and Structural Developments of High Purity Al and Al-Cu-Mg-Zr Aluminium Alloy. In.: *Aluminium Alloys, Theory and Applications,* edited by T. Kvačkaj, R. Bidulský, InTech, Rijeka, 2011, p. 3-26, doi:10.5772/14425
[8] A. Zeleňák et al.: *Microhardness of sintered PM materials prepared from coated powders.* In.: Vrstvy a povlaky 2012, Rožnov pod Radhoštěm, Miloš Vavrík/Trenčín, 2012, p. 141-144
[9] K. Sangwal: Materials Chemistry and Physics, Vol. 63, 2000, No. 2, p. 145-152, doi: 10.1016/S0254-0584(99)00216-3

DOI 10.12776/ams.v22i3.758 p-ISSN 1335-1532
e-ISSN 1338-1156

[10] K. Sangwal, B. Surowska, P. Błaziak: Materials Chemistry and Physics, Vol. 77, 2002, No. 2, p. 511-520, doi: 10.1016/S0254-0584(02)00086-X
[11] J. Gong, J. Wu, Zh. Guan: Journal of the European Ceramic Society, Vol. 19, 1999, No. 15, p. 2625-2631
[12] X. J. Ren, R. M. Hooper, C. Griffiths: Journal of Materials Science Letters, Vol. 22, 2003, No. 15, p. 1105-1106, doi: 10.1023/A:1024947210604
[13] V. Navrátil, J. Novotná: Journal of Applied Mathematics, Vol. 2, 2009, No. 3, p. 241-244
[14] JCMG 200:2012 International Vocabulary of Metrology – Basic and General Concepts and Associated Terms, 3rd edition
[15] OIML D10:2007 Guidelines for the determination of calibration intervals of measuring Instruments
[16] M. Down et al.: *Measurement system analysis,* fourth ed, Chrysler Group LLC, Ford Motor Company, General Motors Corporation, 2010
[17] STN EN ISO 6507-2:2005 Metallic materials. Vickers hardness test. Part 2: Verification and calibration of testing machines
[18] STN EN ISO 6507-1:2005 Metallic materials. Vickers hardness test. Part 1: Test method
[19] J. Petrík, P. Bella, A. Guzanová, I. Sinaiová: Annals of the Faculty of Engineering Hunedoara - Journal of Engineering. Vol. 12, 2014, No. 3, p. 239-242
[20] R. Machaka et al: Advances in Materials Science and Engineering, Vol. 2016, 2016, article ID 539252, 6 pages, doi:10.1155/2011/539252
[21] M. Kupková et al.: *Hardness and indentation size effect in PM materials prepared from coated powders.* In.: Vrstvy a povlaky 2011, Rožnov pod Radhoštěm, Miloš Vavrík/Trenčín, 2011, p. 95-100
[22] K. Lestyánszka-Škůrková, J. Kudičová: *Determining the total dispersion zone for micrometer SM measuring equipment*. In: Research papers Faculty of Materials Science and Technology Slovak University of Technology in Trnava. Vol. 19, 2011, No. 30, p. 45-50
[23] *Methodical guidelines for accreditation. Proficiency testing schemes. MSA 0113-98.* Bratislava. SNAS, 1998, (in Slovak)
[24] R. Palenčár et al.: *Statistical methods in metrological and testing laboratories,* First ed., Grafické štúdio Ing. Peter Juriga, Bratislava 2001, (in Slovak)
[25] [1.9.2007], http://rimarcik.com/navigator/metody.html
[26] J. Petrík, P. Palfy: MAPAN – Journal of Metrology Society of India, Vol. 29, No. 1, 2014, pp. 43-50, doi:10.1007/s12647-013-0062-0
[27] J. Petrík: *The Indentation Size Effect (ISE) of metal*. In: Proceedings of 5th Conference "StrojírenskáTechnologie", Plzeň, January 22-23, 2013, p. 43-49

Acknowledgement
Authors are grateful for the support of experimental works by the Scientific Grant Agency of the Ministry of Education of the Slovak Republic No. VEGA 1/0173/14.