THE INFLUENCE OF THE LOAD ON THE QUALITY OF MICROHARDNESS MEASUREMENT

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Abstract
The objective of submitted work is to analyze the influence of the load on the micro-hardness of Cu, Al, Co, Ni, Fe, Zn and glass. The results were validated by Measurement Systems Analysis (MSA), Analysis of Variance (ANOVA) and Z-score. The relationship between the load and micro-hardness for measured materials can not be explained by Kick's Law (the value of Meyer's index "n" is different from 2). The micro-hardness increases with increasing load up to 0.2942 N; the reverse ISE behavior with n >2 is typical for this load interval. The influence of the load on the micro-hardness is statistically significant. The uncertainty decreases with increasing load and micro-hardness and of material. The capability of measurement process increases with increasing micro-hardness or Meyer's index "n". All results of Z-score are satisfactory.

Keywords: Hardness test, microindentation, metal

1 Introduction

The Vickers test is the standard method for measuring the hardness of metals, particularly those with extremely hard surfaces: the surface is subjected to a standard pressure for a standard length of time by means of a pyramid-shaped diamond with vertex angle 136°. The diagonal of the resulting indention is measured under a microscope. The Vickers testing method is accurate and sensitive hardness test method. Thoroughly prepared surface before test is required. Micro-hardness method is frequently used for determination of hardness of small items or thin layers, and identification of individual phases in metallography. The principle of measurement is identical to Vickers method, except for considerably smaller loads (or test forces). Like in any test of mechanical properties, there is obvious requirement for reliability of measurement results, which is unthinkable without sufficient quality of measurement process. New indentation technique, depth sensing indentation, allows wider information extraction from measured data [1].

In contrast to conventional mechanical testing the stress/strain field beneath the contact point of indentation impression is crucially complicated. Gilman [2] addressed in his article that "Hardness measurements are at once among the most maligned and the most magnificent of physical measurements". Maligned because they are often misinterpreted by the uninitiated, and magnificent because they are so efficient in generating information for the skilled practitioner.

Due to large elastic moduli, the contact deformation of ductile metals is predominantly out of elastic regime, yielding significant plastic flows and leaving a finite residual impression after

unload. On the basis of the experimental results for spherical indentation on various metallic alloys, along with the considerations on the geometrical similarity of contact, in 1908 Meyer proposed the concept of contact hardness as a mean contact pressure defined by the applied indentation load divided by the projected area of residual impression [3].
The advantage of Vickers test is (macro)hardness independence (by definition) on load, because indentations with various diagonals are geometrically even [4]. The stability of (macro)hardness value despite of force change is confirmed by Kick's Law – the applied load and impression diagonal yields a constant value for Mayer's index n =2. The (macro)hardness should be independent of indentation size. However, the most important and intractable problem associated with Vickers hardness testing at low loads, i. e. micro-indentation hardness testing (with the indentations less than about 10 μm deep as a rule [5]), is that concerned with change in indentation size, namely the indentation size effect (ISE) [6]. The micro-hardness of solids vice-versa depends on the applied load. This phenomenon usually involves a decrease in the apparent micro-hardness with increasing test load, i.e., with increasing indentation size [7]. The study of relationship between micro-hardness and load is carried out not only for metallic materials, but also for various tin films deposited by evaporation or produced plasma deposition, semiconductors, organic crystals and even for brushite ($CaHPO_4.2H_2O$) occuring in kidney stones [8-10].
The aim of submitted work is to study the dependence of the micro-hardness of six metals and of the glass on the applied loads ranging from 0.09807 N to 0.9807 N. The results were evaluated by Measurement Systems Analysis (MSA), using GRR method (analysis of repeatability and reproducibility), one factor analysis of variance (ANOVA) and Z-score. In addition, the present study also discusses the relationship between Mayer's index and uncertainty of measurement or indices of measurement process capability.

2 Experimental materials and procedures

Six metals and the glass were used as experimental materials: 1. copper for semiconductors (Cu ≥ 99.99 %), 2. cast electro-conducting aluminum (Al ≥ 99.5 %), 3. annealed iron (ferrite), 4. electrolytic nickel (Ni ≥ 99.93 %), 5. electrolytic cobalt (Co ≥ 99.60 %), 6. cast zinc (98.05 % Zn, 0.42 % Fe, the micro-hardness of zinc matrix was measured out of the intermetalic phases Fe_3Zn_{10}, Al_5Fe_5, $ZnFe_2O_4$ and $Fe_{11}Zn_{40}$ confirmed by metallographic and X-ray analysis).

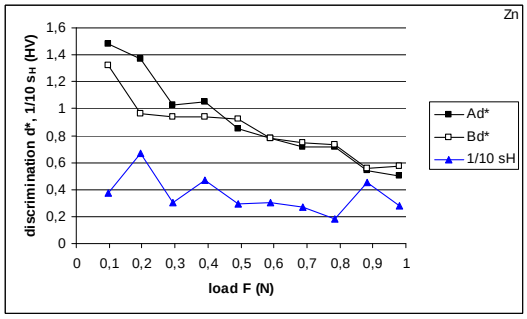

Fig.1 The relationship between discrimination d* and standard deviation s_H for Zn.

The surface of the metals was wet ground using silicon carbide papers (the sequence 220, 240… and 3000 ANSI/CAMI grit), mechanically polished using water suspension Al_2O_3 and

ultrasonically cleaned. The grain size was determined according to standard STN 42 0462 by grain counting method, **Tab. 1**.

Table 1 The diameter of the grains in tested metals.

Metal	Cu	Al	Co	Ni	Co	Fe	Zn
diameter of grains	8 ÷ 15 mm	90 ÷ 170 µm	20 ÷ 30 µm	20 ÷ 50 µm	20 ÷ 30 µm	34 ÷ 39 µm	200 ÷ 300 µm

The micro-hardness of flat glass thick 5 mm with homogeneous structure without visible grains was measured on the original surface. The glass specimen needed no such preparation, for its surface is mirror smooth as a result of the fabrication history.

The Vickers micro-hardness tester is not legal measuring instrument (Act No. 142/2000 Coll.), metrological confirmation is limited to direct or indirect calibration. Metrological confirmation shall be designed and implemented to ensure that the metrological characteristics of the measuring equipment satisfy the metrological requirements for the measurement process [11].

The micro-hardness tester Hanemann, type Mod D32, a part of optical microscope NEOPHOT 32 was the measuring equipment. The magnification of indentation measuring device is 480×. The tester was indirectly calibrated according to standard [12]. The results of calibration were used for calculation of relative expanded uncertainty U_{rel} of the values of hardness according to standard [13]. The load of calibration F = 0.4903 N, application time 15 seconds, the specified hardness of used reference block – certified reference material (CRM) H_c = 195 HV0.05, the uncertainty of CRM u_{CRM} = 4.0 HV0.05. The result of calibration: average measured hardness $\bar{H}$ = 197.87 HV0.05, u_H = 2.594 HV0.05, the repeatability of tester r_{rel} = 2.82 %, relative maximum error E_{rel} = 1.48 % and relative maximum permissible error of the tester (relative expanded uncertainty) U_{rel} = 6.63 %. The tester satisfied the requirements of the standard.

It is necessary to remember the fact that indirect calibration of micro-hardness testers is not routinely practiced process unlike the (macro)hardness testers. Small dimensions of indentations, especially with irregular shape are measured with difficulty. Small difference in reading of dimension of diagonals has significant effect on the value of micro-hardness and makes possible the influence of individuality and skill of appraiser. Unsatisfactory calibration results could be improved by greater magnification (with demands of the quality of metallographic specimen), selection of appraisers (their competence, including education, preparation and experience), higher quality of CRM (low uncertainty), strict observance of operating instructions (standardized methods), the conditions of environment [14]. It is possible that high value of uncertainty of calibration is a result of low capability (high value of Gauge Repeatability and Reproducibility index %GRR, obtained by MSA analysis) [15].

The linearity of tester was evaluated by software CAQ Palstat. The reference lengths of diagonals, calculated for used CRM and load, were compared with measured values. The bias of linearity is satisfactory only for loads 0.68649, 0.78456 and 0.9807 N.

The measurement carried out one appraiser. One metal was measured two times. Five indentations were made at each load/test force F 0.09807, 0.19610,…0.9807 N. Appraiser performed five indentations (A), evenly distributed around the center of the field of view in accordance with the requirements of the standard for the minimal spacing between the adjacent indentations (3 × the average indentation diagonal). The measurement repeated just the same appraiser (B) near the place of former indentations on the next day. The load application time was 15 seconds. One indentation was made out in the center of grain in random order, only in case of Cu all indentations were in one grain. **Fig. 2** illustrates the relationship between applied load and micro-hardness for tested materials.

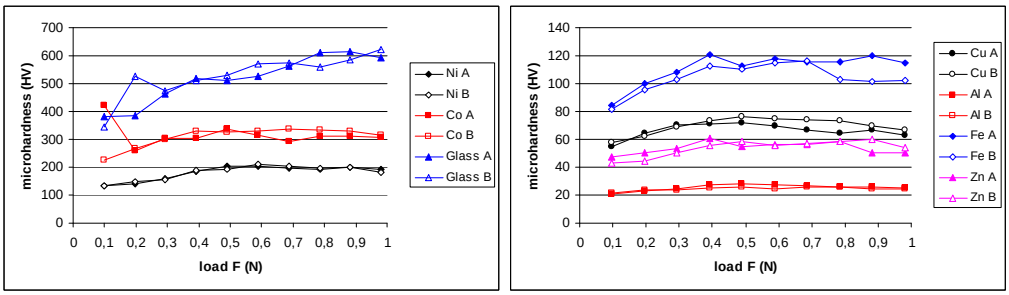

Fig.2 Relationship between applied load and microhardness.

The normality was determined by Freeware Process Capability Calculator software (Anderson – Darling test, p $\geq$ 0.05 for file with normal distribution). The standard methods of MSA assume normal probability distribution. If normality of the file is not confirmed, the error of the measurement system is overestimated [16]. Grubbs' test (significance level α =0.05) was used for detection of statistical outliers. Their presence would indicate measurement process suffering from special disturbances and out of statistical control. The normality and the outliers were determined for files, involving all measurements on one metal by "both" appraisers at all loads (n = 100 indentations). As it can be seen in **Tab. 2**, the normality was confirmed for Cu. A marginal occurrence of outliers verifies that measurement process avoided the gross errors.
A general rule of thumb is that the effective resolution - discrimination d*, the value of the smallest scale division – graduation or the drum step of indentations measuring device, comparable to process variation expressed in standard deviation s_H (both figured in HV) ought to be at most one – tenth [16].

$$d^* = \left| \frac{HV_5 - HV_1}{d_5 - d_1} \right| \tag{1}$$

where d_1 and d_5 are mean values of length of two diagonals of "hardest" (HV_5) and "softest" (HV_1) of 5 indentations. All measurements of micro-hardness do not satisfy the requirement of effective resolution. **Fig. 1** explained "the best" relationship (Zn). The analysis indicated that the increasing load and decreasing hardness results in satisfaction of aforesaid request for discrimination.

Table 2 The average hardness (HV), standard deviation s_H (HV), values p (normality) and outliers.

	$\overline{H}_A$	$\overline{H}_B$	$\overline{H}_{A+B}$	s_H A+B	Normality A+B, p	Outliers A+B
Cu	66.42	69.84	68.13	6.008	0.16089	0
Al	25.52	24.46	24.99	2.203	0.00091	0
Co	316.86	309.70	313.28	39.276	0	0
Ni	182.06	180.88	181.47	25.818	0	0
Fe	111.12	103.98	107.55	11.626	0.00056	0
Zn	54.44	52.86	53.65	5.533	0.00306	1
Glass	516.06	529.7	522.88	82.724	0.00001	0

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

$$z_i = \frac{x_i - \bar{x}}{s} \tag{2}$$

x_i is the hardness of the metal, measured at individual load by one appraiser, $\bar{x}$ is average hardness of the file ($\bar{H}_{A+B}$, tab. 2) and „s“ is standard deviation of the file (s_H A+B, tab. 2). The results $|z_i| \leq 2$ are satisfactory and $|z_i| \geq 3$ are unsatisfactory [17]. The characteristic for lower loads, 0.09807 N and 0.19610 N are conditionally satisfactory or unsatisfactory, whereby the hardness value is significantly low. The hardness of metals is relatively stable at the loads above 0.19610 N, for soft metals (Al and Zn) it gently decreases at loads above 0.78156 N. The glass behaves differently. The micro-hardness evenly increases with increasing load. The difference between the appraisers is minor with the exception of low loads of Co.

One factor analysis of variance (ANOVA) confirms statistically significant influence of the load on hardness of tested materials (tab. 3, the column p HV, the value $p < 0.05$) with the exception of Co.

The relative expanded uncertainty of measured hardness was calculated according to [13]:

$$U = k \cdot \sqrt{u_E^2 + u_{CRM}^2 + u_H^2 + u_{\bar{x}}^2 + u_{ms}^2} \tag{3}$$

$$U_{rel} = \frac{U}{\bar{H}} \cdot 100\% \tag{4}$$

where U is expanded uncertainty, the coverage factor k = 2, the standard uncertainty according to the maximum permissible error u_E = 6.9642 HV0.05, the standard uncertainty of used CRM u_{CRM} = 4.0 HV0.05, the standard uncertainty of hardness testing machine at calibration (proportional to standard deviation of calibration) u_H = 2.594 HV0.05, $u_{\bar{x}}$ is standard uncertainty when measuring a test piece, it is a function of standard deviation s_H A+B (tab. 2), u_{ms} is standard uncertainty of the resolution of the indentations measuring optical system (the values of u_{ms} are 0.4 ÷13.4 HV, increase with increasing hardness and decreasing load), $\bar{H}$ is average hardness $\bar{H}_{A+B}$ in tab. 2. The uncertainty arising from the drift of CRM u_{CRM-D} was ignored, used CRM was calibrated only once.

The uncertainty decreases with increasing both the load and the hardness as it can be seen in fig. 3. One factor analysis of variance (ANOVA) confirms statistically significant influence of the load on relative expanded uncertainty U_{rel} of tested materials (**Tab. 3**, the column p U_{rel}, the value $p < 0.05$) with the exception of glass.

Table 3 The p values for ANOVA and capability indices.

	p HV	p U_{rel}	%R	%X	%EV	%AV	%PV	%GRR
Cu	0.011694	0.000179	0	55	37.0	36.2	85.6	51.8
Al	0.000401	0.014432	0	40	37.4	33.9	86.3	50.4
Co	0.933385	0.002213	0	55	47.8	18.6	85.9	51.2
Ni	1.34E-07	7.35E-06	5	90	32.1	0.0	94.7	32.1
Fe	0.007853	5.17E-05	5	60	35.0	39.8	84.8	53.0
Zn	0.026119	0.002047	0	35	57.0	19.2	79.9	60.2
Glass	0.001339	0.121127	0	60	38.2	10.3	91.8	39.5

Measurement systems analysis (MSA) is an experimental and mathematical method of determining how much the variation within the measurement process contributes to overall

process variability. The measurement process, running in capable measurement system (consists of measurement equipment, samples, environment, method, appraisers…) is capable as well.
The computation of capability by GRR method MSA (analysis of repeatability and reproducibility) was carried out in accordance with [16] with provision for the particularities of the hardness measurement capability [18,19]. The software Palstat CAQ with significance level α = 0.01 and confidence level α = 0.01 (5.15 σ) was used for capability calculation. The values of capability indices, %R, %X, %EV, %AV, %PV and %GRR are in tab. 3.

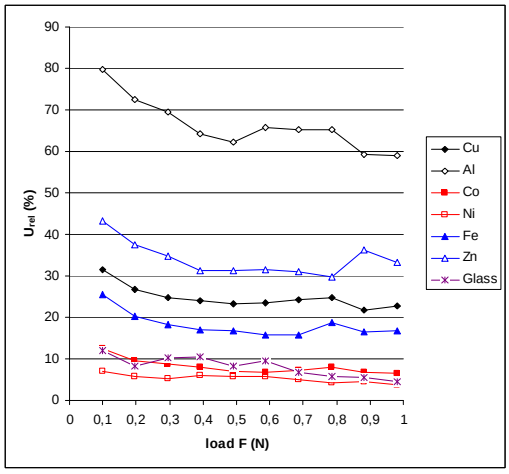

Fig.3 The relationship between the load and relative expanded uncertainty U_{rel}.

The measurement system ought to be in statistical control before capability is assessed. The process is under control, if all ranges are between control limits (UCL – upper/, LCL – lower control limit) of the range control chart. The problems with statistical control were found at Ni and Zn files (tab. 3, column %R, the number presents the measurements out of the control limits in %).
The area within the control limits of the X-bar control chart represents measurement sensitivity („noise“). One half or more of the averages should fall outside the control limits. If the data show this pattern, then the measurement system should be adequate to detect variation between the values of hardness, affected by levels of applied load F. The measurement system can provides useful information for analyzing and controlling the process in that case. Otherwise the measurement system lacks adequate effective resolution (Al and Zn, column %X of tab. 3).

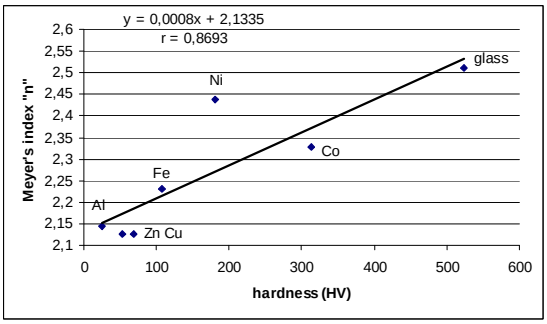

Fig.4 The relationship between the average micro-hardness and Meyer’s index “n” .

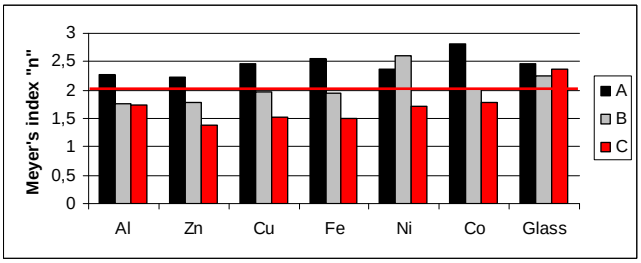

Fig.5 Meyer's index "n" for loads A (0.09807-0.29420 N), B (0.39228-0.58842 N) and C (0.68649-0.98070 N).

The %EV index represents the cumulative influence of measurement equipment, measuring method and environmental conditions on the variability. It is a function of average range of trials of all appraisers.

The %AV index represents the influence of appraisers on the variability, for example their liability, responsibility and competence. It is a function of the maximum average appraiser difference. High value of %AV proves low stability of the appraiser's work quality as compared with %EV.

The %PV is a function of the load range. It is sensitive to variability of applied loads F. The value of %PV indirectly defines suitability of equipment for specific measurement. %PV above 99 % has excessively accurate, above 90 % suitable, above 70 % satisfactory and above 50 % inaccurate equipment [20]. Used tester comes across as satisfactory or suitable.

The %GRR index represents the process capability in practice. For acceptable measurement system %GRR < 10 %, %GRR > 30 % is not acceptable. Analyzed measurement system and also the process carried in it are not acceptable – capable for all tested materials. It is possible, that non - capability is typical for micro-hardness, but also for (macro)hardness measurement [21].

3 Results and discussion

It is well known that the apparent micro-hardness of solids depends on the applied indentation test load. This phenomenon, known as the indentation size effect (ISE) usually involves a decrease in apparent micro-hardness with increasing applied test load F. In order to describe the ISE behavior of materials, several relationships between the applied indentation test load F and indentation diagonal length *d* have been given in the literature. The simplest way to describe the ISE is Meyer's Law

$$F = Ad^n \tag{5}$$

where the exponent "n", the Meyer index (number), is a measure of the ISE and *A* is a constant [7]. **Tab. 4** shows the values of Meyer's index n and lnA for individual appraisers (columns n_A, n_B, $\ln A_A$, $\ln A_B$) and for both appraisers together (columns $n_{(A+B)}$, $\ln A_{(A+B)}$) as a slope (n) and y-intercept (lnA) of a straight line of the linear relationship between the applied load (ln F (g)) and average diagonal of five indentations (ln d (μm)). For ISE behavior, the exponent $n < 2$. When $n = 2$, the hardness is independent of the applied test load and is given by Kick's Law [3,8,10].

Reverse ISE behavior was observed because all measured values of n > 2. The value of n increases with increasing of tested material micro-hardness with strong correlation (r = 0.8693),

fig. 4. Reverse ISE behavior was also obtained when steel CRMs were used as a measured samples [22]. This fact is not in accordance with literary sources as the load – micro-hardness relationship has mostly ISE behavior. Possible sources of this disagreement can be for example friction effects, structural non-uniformity of the deformed volume, mixed elastic and plastic deformation [23], polishing or quality of indenter.
An inverse relationship between lnA and index "n" may be noted. This implies that a metal characterized by a higher index "n" has a lower value of lnA and vice-versa.

Table 4 The indices of ISE for individual files, correlation coefficients r for relationship between ln d and ln F (together results of A and B).

	n_A	n_B	$n_{(A+B)}$	$\ln A_A$	$\ln A_B$	$\ln A_{(A+B)}$	$r_{(A+B)}$
Cu	2.0905	2.1718	2.1261	-3.6596	-3.8927	-3.7587	0.9945
Al	2.1822	2.1146	2.1435	-5.0253	-4.7801	-4.8898	0.9966
Co	2.3089	2.3534	2.3271	-2.7183	-2.7888	-2.7425	0.9934
Ni	2.4363	2.4405	2.4383	-3.6730	-3.6938	-3.6831	0.9940
Fe	2.2877	2.2905	2.2313	-3.7772	-3.5244	-3.6255	0.9937
Zn	2.0390	2.2356	2.1266	-3.6757	-4.4376	-4.0163	0.9930
Glass	2.5547	2.4748	2.5114	-2.7174	-2.4723	-2.5859	0.9940

The values of indices "n", presented by fig. 5 were obtained in the same way as before. The values A were calculated for loads A (0.09807 ÷ 0.29420 N), B (0.39228 ÷ 0.58842 N) and C (0.68649 ÷0.98070 N). The value of index n > 2 in group A for all materials (n = 2.4462 in average). The values of "n" are close to Kick's Law in group B, especially for Co, Cu and Fe (n = 2.0481 in average). The group C contrary to expectation (higher load would to raise the tendency for behavior according to Kick's Law) has ISE behavior (n = 1.7161 in average).
The relationship between Meyer's index "n" and individual indices of capability is in **Fig. 6**. The capability of measurement process increases (The value of %GRR decreases, r = 0.8686) with rising the value of index "n" towards to more reverse ISE behavior (and higher hardness).

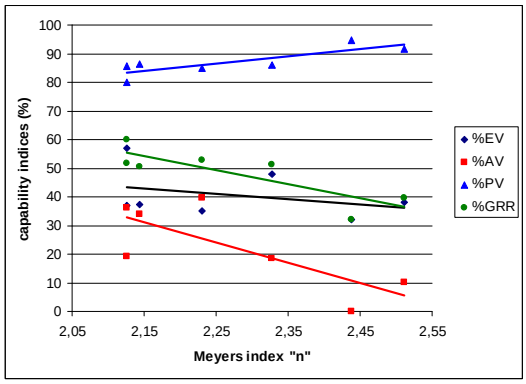

Fig.6 The relationship between Meyer's index "n" and the indices of capability.

4 Conclusions

1. The relationship between the load and micro-hardness for measured materials can not be explained by Kick's Law (the value of Meyer's index "n" differs from 2).

2. The micro-hardness increases with increasing load up to 0.39228 N; the reverse ISE behavior is typical for this load interval.
3. The influence of the load on the micro-hardness is statistically significant for all tested materials except for Co.
4. The uncertainty decreases with increasing both load and micro-hardness of tested material.
5. The capability of measurement process increases with increasing micro-hardness or Meyer's index "n".
6. No unsatisfactory results were identified by Z-score.

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