Jozef Petrík, Vojtech Mikloš, Vojtech Špeťuch, Tomáš Tompoš

Technical University of Košice Faculty of Metallurgy, Slovakia e-mail: jozef.petrik@tuke.sk

THE INFLUENCE OF THE BALL MATERIAL ON THE BRINELL HARDNESS

A Brinell hardness tester with carbide and steel balls was calibrated and thereafter the hardness of ten samples of steel STN 41 1600 was measured by four appraisers. The uncertainty of calibration and of the hardness measurement were calculated. The results obtained with carbide and steel balls were confronted using uncertainty analysis, measurement systems analysis (MSA), analysis of variance (ANOVA), unpaired t-test and Z-score.

The calibrated hardness tester is not considered satisfactory because the values of relative expanded uncertainty of calibration (relative maximum permissible error) and maximum error exceed the values permitted by the standard for both balls. The statistically significant difference between the uncertainties and relative maximum error obtained by carbide and steel balls was not confirmed neither at calibration nor at measurement of steel sample hardness. Measurement system analysis confirmed some influence of used ball materials on the results of individual appraisers on the capability of the hardness measurement process (indices %AV, %GRR and ndc) in accordance with the results of the unpaired t-test.

Keywords: calibration, HBS, HBW, uncertainty, t-test, ANOVA, Z-score

1. INTRODUCTION

The aim of the submitted work is to evaluate the results of a Brinell hardness measurement carried out by standardized tungsten carbide balls (HBW) and by hardened steel balls, nonstandardized at present [1]. The hardened steel balls are frequent accessories of older testers or are used as the indenter from time to time as a result of the appraiser's incompetence.

The Brinell hardness test uses a machine to press a ball into the surface of the test specimen. The machine applies a test force proportional to the ball diameter and tested material. The load is usually applied for 10 to 15 seconds, alternatively 15-180 seconds for soft metals. Among used hardness tests, the Brinell ball makes the deepest and widest indentation, so the test averages the hardness over a wider amount of material, which will more accurately account for multiple grain structures, and any irregularities

in the uniformity of the alloy (typical for cast structure) and soft materials. A wide indentation, on the other hand, can impair the surface of the specimen [2, pp. 299, 319].

Because the hardness tester is not a legal measuring instrument according to Slovak law, its verification is not necessary to ensure relevant regulations (Metrological Act 142/2000 Z. z.). Metrological confirmation is limited only to calibration. The indirect method of calibration (term "verification", used in [3, pp. 6] indicates a legal process according to the aforementioned law) was used by reason of the stiff demands for technical devices and methods in the case of the direct method. The calibration was carried out according to standard STN EN ISO 6506-2 [4].

Two certified reference materials (CRM) in the form of reference block were used as the standards, their specified hardness and uncertainty according to calibration certifications are in Table 1. The force has been applied for 30 seconds.

2. CALCULATION OF UNCERTAINTY ACCORDING TO STANDARD ISO 6506-2

The calibrated hardness tester HPO 250 was made by VEB Werkstoffprüfmaschinen "Fritz Heckert" (former East Germany) in 1982. The magnification of the measuring device is 10×. The test force was 1839 N (187.5 kg) and the ratio test force/ball diameter $\frac{0,102F}{D^2}$ = 30.01 N mm⁻² with the diameter of the used ball 2.5 mm. Four appraisers A, B, C and D measured both perpendicular diameters of 5 indentations, carried out on the relevant standard by the relevant ball.

The value of discrimination (resolution, smallest scale division) δ_{ms} was calculated according to formula 1.

$$\delta_{ms} = \frac{H_{\text{max}} - H_{\text{min}}}{(d_{\text{max}} - d_{\text{min}}) \times 1000} \tag{1}$$

The mean diameter

$$\overline{d} = \frac{d_1 + d_2 + d_3 + d_4 + d_5}{5} \tag{2}$$

The repeatability of tester r_{rel} in specific conditions can be calculated as:

$$R_{rel} = 100 \times \frac{d_5 - d_1}{\overline{d}}\% \tag{3}$$

 d_5 is the indentation with ultimate and d_1 one with minimal diameter.

The repeatability of the tester r_{rel} is considered satisfactory if it satisfies the conditions given in [4, tab. 2, pp. 9] e. i. less than 2 % for the HBS standard and 2.5 % for the HBW standard.

Table 1. Specified hardness and uncertainty of used standards

Stan	dard (CRM)	specified hardness H _c (HB)	U _{CRM} (HB)	U _{CRM} (HB)	
"HBS"	HBS 2.5/187.5	242.2	3.63	1.82	
"HBW"	HBW 2.5/187.5	185.0	3.30	1.65	

The average measured hardness of standard

$$\overline{H} = \frac{H_1 + H_2 + H_3 + H_4 + H_5}{5} \tag{4}$$

The error at specific conditions of calibration:

$$E = \overline{H} - H_c \tag{5}$$

Relative maximum error

$$Erel = 100 \times \frac{\overline{H} - H_c}{H_c} \tag{6}$$

The maximum error of the testing machine, expressed as percentage of the specified hardness of the standard, shall not exceed the values (2 % for the HBS standard and 2.5 % for the HBW one) given in [4, tab. 2, pp. 9]. The values of repeatability, standard deviation and maximum error are presented in Table. 2.

ball Standard	Appraiser	\overline{H}	r _{rel}	\mathbf{S}_{H}	E _{rel}	u _H	u _{ms}	u _{HTM}	U _{HTM}	ΔH_{HTMmax} /H _c
ball	А	257.00	1.43	3.34	6.11	1.75	0.16	2.51	5.02	8.18
HBS	В	254.62	1.74	3.64	5.13	1.87		2.62	5.23	7.29
standard	С	254.84	0.68	1.53	5.22	0.79		1.99	3.98	6.86
HBS	D	256.86	0.21	0.51	6.05	0.26		1.85	3.69	7.57
ball	А	196.04	0.93	1.42	5.97	0.73	0.11	1.81	3.62	7.92
HBW	В	194.27	0.32	1.35	5.01	0.69		1.79	3.59	6.95
standard	C	195.01	0.93	1.40	5.41	0.72		1.80	3.61	7.60
HBW	D	200.90	1.46	2.67	8.59	1.37		2.15	4.30	10.92

Table 2. The values for calculation of the uncertainty according of the calibration

The present calculations supposed that the result of calibration equals its ideal (real) value. But, as results from the uncertainty definition: "Uncertainty is a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" and for this fact must be made provision

The uncertainty of indirect calibration is calculated:

$$u - HTM = \sqrt{u_{CRM}^2 + u_{CRM-D}^2 + u_H^2 + u_{ms}^2}$$
(7)

The standards uncertainties uCRM are in Table. 1. The uncertainty resulting from the standard hardness variation u_{CRM-D} in time (drift) was ignored. It is impossible to determine this value, because both used standards were calibrated only once time (XI/2005 "HBS" and XI/2007 "HBW").

The standard uncertainty of the hardness tester is:

$$u_H = \left[\frac{t \times s_H}{\sqrt{n}}\right] \tag{8}$$

 s_H is standard deviation of the results of indirect calibration, Student's factor t = 1.15 for n = 5 (trials – repeated measurements of one appraiser) and \Box = 0.317 [4, pp. 15]. Standard deviation

$$s_H \sqrt{\frac{1}{n-1} \sum_{i=1}^n \left(H_i - \overline{H}\right)^2} \tag{9}$$

The uncertainty of the measuring device from this source -ums arises from the inaccuracy of the device measuring the diameters of indentations.

$$u_{ms} = \frac{\delta_{ms}}{2\sqrt{3}} = 0.29 \times \delta_{ms}[1, \text{pp.17(tab.C1, step7)}]$$
(10)

 δ_{ms} is the hardness value of the discrimination of the tester at used magnification (0.552 HBS for the steel ball and standard HBS and 0.379 HBW for the carbide ball and standard HBW) calculated according to equation (1). The error of calibration

$$\overline{b} = \overline{H} - H_c = E \tag{11}$$

The maximum permissible error of the tester including the measurement uncertainty:

$$\Delta H_{HTMmax} = U_{HTM} + |\overline{b}| \tag{12}$$

The expanded uncertainty $U_{HTM} = \mathbf{k} \times u_{HTM}$ (13)

The coverage factor k = 2

Relative maximum permissible error of the tester (relative expanded uncertainty):

$$U_{rel} \frac{\Delta H_{HTMmax}}{\overline{H}} \tag{14}$$

This value is not considered satisfactory unless it satisfies the conditions given in [4, pp. 9], i.e. up to ± 2 % for the standards with specified hardness above 225 HBS (used HBS standard) and up to ± 2.5 % for the standards up to 225 HBS (used HBW standard).

The repeatability is satisfied for all appraisers and both ball materials. As far as E_{rel} (relative maximum error), the tester was not satisfying for all balls and appraisers. The average E_{rel} was 6.24 % for HBW and 5.63 % for HBS. According to single factor analysis (ANOVA) this difference is not statistically significant (p = 0.493753), the part of the variability explained by ball materials is 8.13 %. According to two-factor analysis (ANOVA) without replication the influences of appraisers (p = 0.240417) and ball materials (p = 0.409183) on the E_{rel} are not statistically significant as well.

The tester was not satisfying for all balls and appraisers in respect of its relative U_{rel} (maximum permissible error or relative expanded uncertainty). The average uncertainty was 8.29 % for HBW and 7.48 % for HBS, better results were surprisingly obtained with non – standardized balls. According to single factor analysis (ANOVA) this difference is not statistically significant (p = 0.421208) for balls, the part of the variability explained by ball materials is 11.0 %. According to two-factor analysis (ANOVA) without replication the influences of appraisers (p = 0.403518) and ball materials (p = 0.417701) are not statistically significant [5, pp. 129].

3. MEASUREMENT OF STEEL HARDNESS

The investigated material was rolled wrought steel STN 41 1600 (equivalent to material E335GC according to standard EN 10025A1). Ten samples $(100 \times 30 \times 5 \text{ mm})$ were evenly cut from the hoop steel. The surface of samples was ground with abrasive papers No. 220, 240, 280, 400 and 500. The microstructure is pearlitic with low ferrite content (Fig. 1). The difference in microstructure of individual samples was negligible. The hardness of the samples was measured as well as calibration (HBW 2.5/187.5 and HBS 2.5/187.5, four appraisers, three trials on each sample, ten samples).



Fig. 1. Microstructure of experimental material (1% nital).

The first step of analysis is to estimate whether the discrimination of the measuring equipment is sufficient. A general rule of thumb is that the discrimination ought to be at least one – tenth the process variation (sx) [6, pp. 44, 74]. Looking at Table 3 we can see that the tester satisfies this condition for all balls and appraisers.

Grubbs' test (with significant level $\Box = 0.05$) detected two outliers (both appraiser D, carbide and steel balls). The statistical outliers would indicate that the process is suffering from special disturbances and is out of statistical control.

The normality was estimated by Freeware Process Capability Calculator software, using the Anderson – Darling test (significance level \Box = 0.05). The value p for the files with normal distribution is more than 0.07 (P – "pass", or F – "fail" when the file has not a normal distribution in Table. 3). The standard methods of MSA assume normal probability distribution. Therefore, before use, the data should be checked to confirm that their distribution is approximately normal. Whereas normality of all the analyzed files was not confirmed, the measurement system error is up to a certain point overestimated [6, pp. 48].

Method	appraiser	Average	s _x	δ_{ms}	Normality	Outliers
HBW	А	211.2	4.122	0.42	P, p: 0.145030	0
	В	210.7	5.542	0.426	P, p: 0.410633	0
	С	212.0	13.954	0.396	F, p: 0.000160	0
	D	214.4	12.998	0.467	F, p: 0.039990	1
HBS	А	211.5	15.078	0.433	F, p: 0.001661	0
	В	208.2	6.321	0.417	P, p: 0.609697	0
	С	223.9	15.922	0.485	F, p: 0.035253	0
	D	201.4	15.204	0.42	F, p: 0.000070	1

Table 3. The values of steel hardness for all ten samples.

The uncertainties of the hardness of individual samples were calculated according to [1, pp. 15], method "without deviation" (method M1), which can be used without making provision for the bias of the hardness tester.

$$U = \mathbf{k} \times \sqrt{u_E^2 + u_{CRM}^2 + u_H^2 + u_H^2 + u_{ms}^2}$$
(15)

The coverage factor k = 2

The standard uncertainty of the maximal permissible error of the used tester

$$u_E = \frac{u_{E2r} \times H_c}{2.8} = 1.65$$
 HBW and 1.73 HBS (16)

 H_c is the specified hardness of the standard (Tab. 1), u_{E2r} is the maximum error of the testing machine expressed as a decimal number (0.025 ≈ 2.5 % for HBW and 0.02 ≈ 2 % for HBS given in [4, tab. 2, pp. 9]).

Standard uncertainties u_H of the hardness tester according to equation (9) for both ball materials and individual appraisers are in Table 2.

The standard uncertainties of the hardness measurement on individual sample

$$u_{\overline{x}} = \frac{t \times s_x}{\sqrt{n}} \tag{17}$$

 s_x is the standard deviation of the 3 trials of one appraiser on one sample (Tab. 3), Student's factor t = 1.3187 for n = 3 and \Box = 0.317 [1, pp. 17]. The values of $u_{\overline{x}}$ are in Table. 4.

Table 4. The values of the standard uncertainty of the hardness measurement on an individual sample $u_{\overline{\tau}}$.

method	Appraiser	1	2	3	4	5	6	7	8	9	10
HBW	А	1.587	2.329	1.587	0.762	0.440	0.0	2.329	0.440	2.641	1.525
	В	1.760	1.760	6.513	4.198	3.603	0.880	0.440	1.320	2.450	1.587
	С	5.076	1.760	0.440	8.835	2.641	5.619	11.818	11.965	1.164	0.440
	D	2.677	6.917	4.901	4.841	16.915	4.901	14.309	0.880	4.198	18.817
HBS	А	2.677	4.224	1.918	2.287	1.760	1.760	6.987	12.323	13.729	19.540
	В	2.687	2.886	3.081	0.440	1.164	1.164	1.164	0.440	2.450	4.999
	С	2.287	2.017	0.880	6.917	4.401	5.076	8.835	1.760	2.017	2.677
	D	2.886	0.440	2.017	2.677	3.521	1.320	13.884	3.080	2.912	13.400

Another source of uncertainty is the measuring device. The uncertainty from this source – ums arises from inaccuracy of the device measuring the diameters of *ums* indentations. The values of ums were calculated according to equation (10), using values of δ_{ms} from Table. The values of the hardness, measured by all appraisers with both balls on individual steel samples are in Figure 2.



Fig. 2. Values of the hardness of the steel samples.

The values of relative expanded uncertainty U_{rel} for individual samples, appraisers and balls can be seen in Fig. 3.



Fig. 3. Values of relative expanded uncertainty U_{rel} for individual samples, appraisers and balls.

The average relative expanded uncertainty U_{rel} was 5.15 % for HBW and 5.28 % for HBS for all appraisers and all samples. According to single factor analysis (ANOVA) the influence of ball material on this difference is not statistically significant (p = 0.927402), the part of the variability explained by ball materials is 0.15 %. According to two-factor analysis (ANOVA) without replication the influences of appraisers (p = 0.538677) and ball materials (p = 0.932317) on U_{rel} are not statistically significant.

The influence of the samples on the uncertainty according to single factor analysis is not closely statistically significant (p = 0.05201), the part of the variability explained by variability between samples is 72.8 % [5, pp. 125].

4. CALCULATION OF HARDNESS MEASUREMENT PROCESS CAPABILITY BY MSA

The Average and Range method (GRR), one of Measurement system analysis (MSA) techniques is an experimental and mathematical method of determining measurement repeatability and reproducibility. It was used for evaluation of the aforesaid measurement process. The computation of capability indices was carried out according to [6, pp. 102-120][7, pp. 142-154]. The software Palstat CAQ with significance level $\Box = 0.01$ % and confidence level $\Box = 0.01$ (5.15 \Box) was used for capability calculation.

ball	%EV	%AV	%PV	%GRR	ndc	%R (appraisers)	%X
HBW	79.9	16.3	57.9	81.5	1.002	12.5 % (C,D)	5.0 %
HBS	50.7	75.3	41.9	90.8	0.652	10.0 % (A,D)	40.0 %

Table 5. The capability indices.

The number of samples and trials depends upon the significance of the characteristic being measured and upon the confidence level required in the estimate of measurement system variation. As with any statistical technique, the larger the sample size, the less the sampling variation and the resultant risk will be present. As a rule, 10 samples, 3 trials and 2 appraisers are used for tests. If possible, the appraisers who normally use the measurement equipment should be included in the study.

The measurement system ought to be in statistical control before capability is assessed, the range (R) control chart is used. The process is in control if all ranges are between control limits. This condition was not satisfied (Tab. 5) for both balls.

The number of distinct categories ("ndc", based on Wheeler's discrimination ratio) is connected with the question of resolution of the measurement equipment. The "ndc" is greater than or equal to 5 for capable processes, results with "ndc" values between 2-5 may be conditionally used for rough estimations. The "ndc" value is unsatisfactory for all appraisers.

The area within the control limits of the X-bar control chart represents measurement sensitivity ("noise"). Since measurements used in the study represent the process variation, approximately 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 sample-to – sample variation and the measurement system can provide useful information for controlling the process. If less than half fall outside the control limits then either the measurement system lacks adequate effective resolution or the sample does not represent the expected process variation. As can be seen in Table 5 the condition of sensitivity was not satisfied.

The %EV index represents the cumulative influence of measurement equipment, the measuring method used and environmental conditions on variability. It is a function of the average range of trials of all appraisers. Contrary to expectation, the use of steel balls resulted in a low (better) value of %EV.

The %AV index represents the influence of appraisers on variability, for example their competence, perceptions, skills, discipline and vigilance. It is function of individual appraisers average values. The value of %AV is higher steel ball.

The %PV index is a function of range of average hardness of individual samples. It is sensitive to the variability between measured samples. Its value indirectly defines the property of equipment used for measurement. The value of %PV above 99 % is for exceedingly accurate equipment, above 90 % for suitable, above 70 % for satisfactory and above 50% for inaccurate one. Equipment with a value up to 50 % is unsuitable [8, pp. 29].

The analyzed process is not capable for both balls, as the value of %GRR (the rate of the manufacturing production process variability that is "consumed" by measurement system variation) is above 10%. The capability of HBW measurement is better than that of HBS. It is possible that the high value of uncertainty is the result of low capability, typical for the hardness testers [9]. For example the %GRR index varied between 40.9 % and 86.5 % at repeated hardness (HBS 2.5/187.5) measurements of steel (STN 41 1373) [10]. The hardness (HBS 5/250) measurement process of Cu – Zn – Al brass castings [11] has also low capability(between 63.7% and 89.4%).

The unpaired t-test was used for comparison two means – the average hardness of all samples measured by steel or carbide balls by individual appraisers. The two – tailed p values for appraiser A (0.9259) and B (0.1135) verify that this difference is not statistically significant, but for appraisers C (0.0096) and D (0.0007) it is.

Measurement system analysis confirmed the influence of the used ball materials on the capability of the hardness measurement process.



Fig. 4. Z-score.

5. CALCULATION OF Z-score

Z-score, routinely applied in interlaboratory comparisons was used for validation of above mentioned results. The value for an individual sample is:

$$z_i = \frac{x_i - \overline{x}}{s} \tag{18}$$

 x_i is the average hardness of one sample, measured by one appraiser with one ball, \overline{x} is the average hardness on one sample measured by all appraisers with all balls, and "s" is standard deviation of the hardness of all samples measured by all appraisers with all balls. Results $-z_i \le 2$ are satisfactory and $-z_i \ge 3$ are unsatisfactory [12, pp. 32][13, pp. 217]. As can be seen in Fig. 4, there are some difference between Z-scores of the results obtained with carbide and steel balls.

6. CONCLUSIONS

1. The calibrated hardness tester is not considered satisfactory because the values of relative expanded uncertainty of calibration (relative maximum permissible error) and the relative maximum error exceed the values permitted by the standard for both balls.

2. The statistically significant difference between the uncertainties and relative maximum error obtained by carbide and steel balls was not confirmed neither at calibration nor at measurement of steel samples hardness.

3. Measurement system analysis confirmed some influence of used ball materials for the results of individual appraisers and on the capability of hardness measurement process

ACKNOWLEDGEMENTS

This work was supported by the Slovak Grant Agency for Science VEGA 1/4141/07.

REFERENCES

- 1. STN EN ISO 6506-1:2005. "Metallic materials. Brinell hardness test". Part 1: Test method.
- 2. Veles, P.: Mechanick'e vlastnosti a skúšanie kovov. SNTL Praha/Bratislava 1985. (in Slovak).
- 3. STN EN ISO 6507-2:2005. "Metallic materials. Vickers hardness test". Part 2: Verification and calibration of testing machines.
- 4. STN EN ISO 6506-2:2005. "Metallic materials. Brinell hardness test". Part 2: Verification and calibration of testing machines.
- 5. Chajdiak, J.: Štatistika v exceli. Statis Bratislava 2002. ISBN 80-85659-27-1. (in Slovak).
- 6. Measurement systems analysis (MSA). Reference manual. Third edition. 2003.
- 7. Plura, J.: *Plànovanì a neustálé zlepšovaní jakosti*. Computer Press. Praha 2001. ISBN 80-7226-543-1.(in Czech).
- Meloun, M., Militký, J.: Kompendium statistického zpracování dat. 1. vydanie. Praha: Academia, 2002. ISBN 80-200-1008-4. (in Czech).
- 9. Tobolski, E.: "Uncertainty in Hardness testing". (part 2). Advanced materials & processes. vol 161, no. 5, 2003, pp. 25.
- Petrík, J., Špet'uch, V.: "The Dependence of Hardness Measurement System Capability on the Time". Transactions of the Universities of Košice, vol. 15, no 3, 2005, pp. 27-33, ISSN 1335-2334.
- 11. Petrík, J., Špet'uch, V.: "The dependence of brass hardness measurement system on the time". *Metrology and measurement systems*. vol. 14, no. 4 ,2007, pp. 595-602. ISSN 0860-8229.
- 12. *Mthodical guidelines for accreditation. Prificiency testing schemes.* MSA 0113-98. Bratislava. SNAS, 1998. (in Slovak).
- Palenčár, R. et al.: Štatistické metódy v metrologických a skúšobných laboratóriách. Bratislava. Grafické štúdio Ing. Peter Juriga, 2001, ISBN 80-968449-3-8. (in Slovak).