THE QUALITY OF HARDNESS TESTER CALIBRATION

JOZEF PETRIK, PAVOL PALFY

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 the most accurate and sensitive hardness test method. It is unsuitable for inhomogeneous and coarse-grained materials. Thoroughly prepared surface before test is required. The Vickers test does not deteriorate the surface of final product as much as Brinell test.

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.

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. Metrological confirmation comprises measuring equipment calibration and measuring equipment verification [1].

The indirect calibration of the hardness tester is checking of a measuring instrument against an accurate CRM (certified reference material, standard) to determine any deviation and correct for errors [2]. The direct calibration includes verification of test force, indenter dimensions (deviation of the angle between opposite faces of the pyramid α), measuring microscope (device for measuring of indentations) and the testing cycle (speed of penetration speed of the indenter, application time and force duration) [3].

The perfect measurement cannot be performed because the values are, by nature, indeterminable. In fact, says the International Organization for Standardization (ISO), it is impossible to fully describe the measured value without an infinite amount of information. In other words, the final corrected result of a measurement is, at best, an estimate of the true value of the quantity that someone intended to measure. The measurement uncertainty is a parameter that characterizes the dispersion of the values that could reasonably be attributed to the measured value [4].

ISSN 1335-1745

A calibration laboratory, or a testing laboratory performing its own calibrations, shall have and shall apply a procedure to estimate the uncertainty of measurement for all calibrations and types of calibrations. Testing laboratories shall have and shall apply procedures for estimating uncertainty of measurement or calibration. When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation, shall be taken into account using appropriate methods of analysis [5].

For indirect calibration of hardness tester against CRM according to the respective standard usually there is not a problem to keep the requirements for repeatability r_{rel} and maximum relative error E_{rel} of the tester. The problem arises after the determination of the maximum permissible deviation of the tester including its measurement uncertainty, which is equivalent to relative expanded uncertainty of calibration U_{rel}, whereas it is frequently higher than the value permitted by the standard. In such case, the measuring device is nonconforming and shall be removed from service.

Table 1 – The results of indirect calibration

No.	Appraiser A						Appraiser B					
	$\overline{H}$ (HV10)	s_H (HV10)	r_{rel} (%)	E_{rel} (%)	u_{ms} (HV10)	U_{rel} (%)	$\overline{H}$ (HV10)	s_H (HV10)	r_{rel} (%)	E_{rel} (%)	u_{ms} (HV10)	U_{rel} (%)
1	506	2.91	0.65	7.16	0.764	8.66	502	7.34	1.69	6.28	0.755	8.34
2	502	5.58	1.30	6.22	0.754	8.08	506	3.86	0.91	7.05	0.763	8.63
3	500	4.53	1.17	5.89	0.751	7.69	504	4.56	1.04	6.72	0.760	8.41
4	500	3.31	0.91	5.94	0.751	7.64	504	2.45	0.65	6.77	0.760	8.30
5	493	6.98	1.80	4.31	0.734	6.57	506	6.32	1.31	7.06	0.763	8.88
6	503	11.75	2.86	6.42	0.756	9.09	499	3.72	0.78	5.56	0.747	7.33
7	493	3.95	0.90	4.26	0.734	6.19	494	1.89	0.52	4.62	0.738	6.40
8	490	3.44	0.90	3.77	0.728	5.72	494	2.29	0.65	4.63	0.738	6.42
9	490	3.62	0.77	3.67	0.727	5.63	495	5,05	1.41	4.85	0.740	6.82
10	490	1.91	0.51	3.67	0.727	5.53	500	4.67	1.17	5.89	0.751	7.70

2 METHODOLOGY

The calibration was realized by two alternating approximately equally skilled appraisers (A, B). The measurement points were along the diameter (rim to rim) of the CRM in equidistant intervals. Appraiser A performed a calibration (5 indentations) followed by appraiser B. The indentations of both appraisers were evenly distributed around the center of the filed of view complying with the standard's requirement for the minimal spacing between the adjacent indentations (3 × the average indentation diagonal) [6]. The force application time was 10 seconds. The values of average hardness and standard deviation s_H for individual calibrations are in tab. 1.

The hardness tester is not legal measuring equipment (Slovak regulation No. 210/2000) and metrological confirmation is limited only to calibration according to standard [3]. Calibrated tester HPO 250 was made by VEB Werkstoffprüfmaschinen „Fritz Heckert“ (East Germany) in 1982. The magnification of measuring device is 140×.

Table 2 – The sensitivity coefficients

appraiser	A			B		
No.	A_F mm^{-2}	A_A $Nmm^2/°$	A_d Nmm^{-3}	A_F mm^{-2}	A_A $Nmm^2/°$	A_d Nmm^{-3}
1	5.17232	102.2656	5289.815	5.129913	101.4271	5224.604
2	5.12696	101.3688	5220.241	5.167001	102.1604	5281.611
3	5.11088	101.0507	5195.754	5.150926	101.8426	5256.945
4	5.11341	101.1009	5199.683	5.15343	101.8921	5260.872
5	5.03512	99.55295	5080.485	5.167307	102.1664	5281.925
6	5.13626	101.5526	5233.794	5.094907	100.735	5171.462
7	5.03211	99.49343	5064.395	5.050165	99.85033	5103.553
8	5.00881	99.03271	5040.943	5.050185	99.85073	5103.574
9	5.00377	98.93126	5033.191	5.06088	100.0622	5119.668
10	5.00356	98.92891	5033.072	5.110889	101.0509	5195.766

The test force/load F = 98.07 N (10 kg). According the direct calibration (VI/08) the deviation for test force 98.07 N is -0.2 % and the deviation of the measurement device is -0.2 % for 0.1 mm and -0.2 % for 0.2 mm. The certified reference material (CRM) in form of hardness reference block with specified hardness H_c = 472.4 HV 10 and expanded uncertainty U_{CRM} = ± 9.448 HV 10 (coverage factor k = 2) was used as a standard. The ambient temperature was 20°C, relative humidity 48 %.

Table 3 – The values of standard uncertainty for deviations of vertex angle α

α (°)	0	0.086	0.0925	0.099	0.1	0.2	0.3	0.4	0.5
u_A (°)	0	0.049652	0.053405	0.057158	0.057735	0.11547	0.173205	0.23094	0.288675

The first step of analysis is to estimate whether the discrimination (effective resolution) d*- the value in HV 10 of the smallest scale division (graduation) of measurement equipment is sufficient. A general rule of thumb is that the discrimination ought to be at least one - tenth the process variation (standard deviation s_H in tab. 1). The discrimination d* = 2.72 HV10 is not sufficient [7].

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Grubbs' test (with significance level $\alpha = 0.05$) detected one outlier (appraiser A, calibration No. 6). The statistical outliers would indicate that the process is suffering from special disturbances and is out of statistical control.

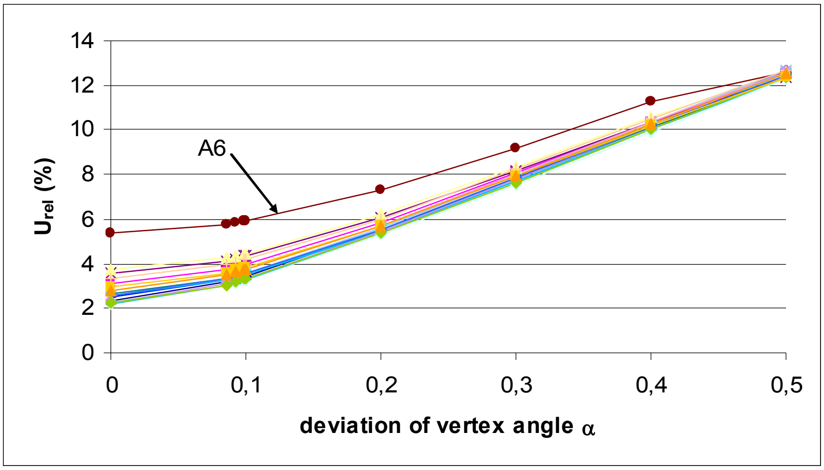

Figure 1 – The influence of vertex angle deviation α on U_{rel} for individual calibrations (ΔF = - 0.2 %, u_{CRM} = 4.724 HV10)

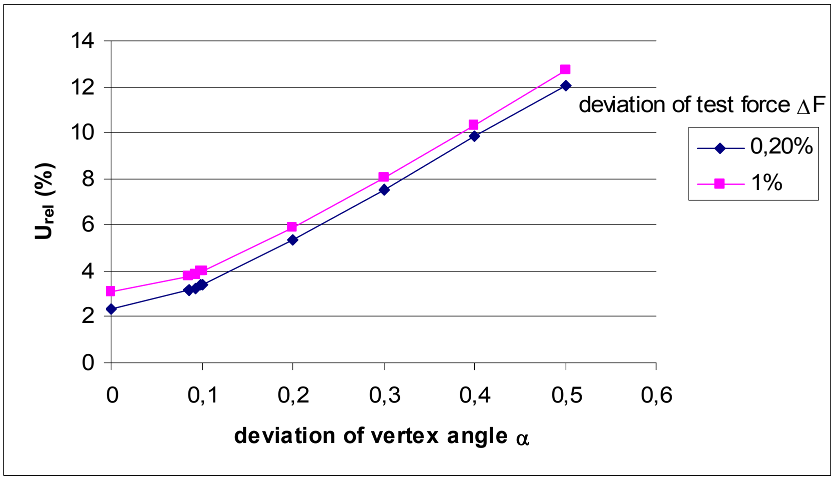

Figure 2 – The influence of vertex angle deviation α and test force deviation ΔF on mean values of U_{rel} (u_{CRM} = 4.724 HV10)

2.1 The indirect calibration

The repeatability of tester $r_{rel} = 100\times\frac{d_5 - d_1}{\overline{d}}\ \%$ (1)

$\overline{d}$ is the mean, d_5 is the maximum and d_1 is minimum value of indentations diagonals.

The error at specific conditions of calibration $E = \overline{H} - H_c$ (2)

$\overline{H}$ is the average hardness of CRM

Relative maximum error $E_{rel} = 100\times\frac{\overline{H} - H_c}{H_c}\ \%$ (3)

The present calculations supposed, that the result of calibration equals to its ideal (or real) value. But, as it results from the uncertainty definition: "Uncertainty is a parameter associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measured value" and for this fact must be made provision [8]. The uncertainty of indirect calibration:

$$u_{HTM} = \sqrt{u_{CRM}^2 + u_{CRM-D}^2 + u_H^2 + u_{ms}^2} \quad (4)$$

The standard uncertainty of used CRM u_{CRM} = 4.724 HV10. The uncertainty resulted drift of CRM u_{CRM-D} was ignored (used CRM was calibrated only once, XI/2005).

Standard uncertainty of hardness tester $u_H = \frac{t\times s_H}{\sqrt{n}}$ (5)

s_H is standard deviation of the results of calibration, Student's factor t = 1.15 for n = 5 (number of trials in one calibration) and significance level $\alpha = 0.317$ [3].

Another source of uncertainty is measuring device.

$$u_{ms} = \frac{2\overline{H}}{d}\,\frac{\delta_{ms}}{2\sqrt{3}} \quad (6)$$

δ_{ms} = 0.0005 mm is the sensitivity of indentations measuring device at used magnification 140× (the unit of abovementioned discrimination d* is HV0.05!).

$$d = \sqrt{0.1891\times\frac{F}{\overline{H}}} \quad (7)$$

F = test force (N)

The error of calibration $\overline{b} = \overline{H} - H_c = E$ (8)

ISSN 1335-1745

The maximum permissible error of the tester including the measurement expanded uncertainty U_{HTM} (coverage factor k = 2):

$$U_{HTM} = k \times u_{HTM} \quad (9)$$

$$\Delta H_{HTM\max} = U_{HTM} + \left|\bar{b}\right| \quad (10)$$

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

$$U_{rel} \quad \frac{\Delta H_{HTM\max}}{\bar{H}} \times 100\% \quad (11)$$

The values $r_{rel} \leq 2\%$, E_{rel} and $U_{rel} \leq \pm 3$ % for satisfactory tester and used CRM [3]. The values of r_{rel} , s_H and E_{rel} are in the tab. 1. It is possible that high value of uncertainty of calibration is a result of low capability (high value of %GRR) [9] and low resolution of the tester. The tester is not satisfactory for all calibrations and all appraisers with respect to its U_{rel}. The two factor ANOVA with replication was used for evaluation of hardness values. The influences of appraiser (p = 0.000303) and calibration's place (p = 3.16 E-9) are both statistically significant.

Table 4 – The values of average diagonals of indentations d and their standard devitation $\bar{d}_{SD}$

	Appraiser	1	2	3	4	5	6	7	8	9	10
d (mm)	A	0.1914	0.19225	0.19225	0.1925	0.1940	0.1921	0.19405	0.1945	0.1946	0.1946
	B	0.1922	0.1915	0.1918	0.19175	0.1915	0.19285	0.1937	0.1937	0.1935	0.19255
$\bar{d}_{SD}$ ($\times 10^{-4}$ mm)	A	5.48	10.61	8.73	6.37	13.81	22.26	7.79	6.85	7.20	3.79
	B	14.09	7.29	8.73	4.68	11.99	7.20	3.71	4.47	9.84	8.91

2.2 The direct calibration

The standard [5] defines Vickers hardness as a function of the measured value of test force F [N], the vertex angle of diamond α (136°) and the indentation diagonal length d [mm]. The measured values of $\bar{H}$ (tab. 1) = H in the formulas (12) – (15). The sources of uncertainty z and sensitivity coefficients A were identified and calculated according to [10][11][12].

$$HV = 0{,}102 \times \frac{2F\sin\left(\frac{\alpha}{2}\right)}{d^2} \quad (12)$$

The values of sensitivity coefficients for test force A_F, vertex angle of diamond A_A and mean diagonal of indentation A_d are:

$$A_F = \frac{\partial HV}{\partial F} = \frac{HV}{F} \tag{13}$$

$$A_A = \frac{\partial HV}{\partial \alpha} = \frac{HV}{2\tan\left(\frac{\alpha}{2}\right)} \tag{14}$$

$$A_d = \frac{\partial HV}{\partial d} = -2 \times \frac{HV}{d} \tag{15}$$

The values of sensitivity coefficients are in table 2.

The uncertainty of specific source was calculated according to formula u = $\frac{z}{\chi}$ z = the source of uncertainty, χ is the value associated with the relative probability distribution [13].

$$u^2 = A_F^2 u_F^2 + A_A^2 u_A^2 + A_d^2 u_d^2 + u_{CRM}^2 \tag{16}$$

1. The test force meets standard tolerance if maximal permissible error (deviation) ΔF = ± 1 % is not exceeded [6] (i.e. z = 2.942 N). The force deviation of tester ΔF = -0.2 %. $\chi = \sqrt{3}$ for assumed rectangular distribution. The values of uncertainty u_F are 0.113242 N (ΔF = -0.2 %) and 0.566208 N (ΔF = ± 1 %).

Table 5 – The values U_{rel} of indirect calibration

	1	2	3	4	5	6	7	8	9	10
A	3.08	3.62	3.40	3.17	4.06	5.45	3.31	3.23	3.26	3.01
B	4.10	3.24	3.40	3.03	3.80	3.24	2.99	3.04	3.53	3.42

2. The vertex angle of pyramid shaped diamond is 136° ± 0.5° according to standard [6]. The source of uncertainty z_A= 0.0925° for average angle deviation α of used diamond (+0.099° for axis x and +0.086° for axis y, measured by TSK C1700 SD2 contourograph, Department of Biomedical Engineering, Automation and Measurement, Faculty of mechanical engineering, Technical University Košice). Rectangular distribution was assumed, $\chi = \sqrt{3}$. The values of uncertainty for deviations of vertex angle α are in tab. 3.

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3. The standard deviation of individual diagonals d ($\bar{d}_{SD}$, tab. 4) for individual appraiser is source of uncertainty z_d. Because normal distribution was assumed, $\chi = 1$ and therefore $u_d = \bar{d}_{SD}$ [13, pp. 23].

4. The uncertainty u_{CRM} = 0, 1, 2, 3 and 4.724 HV10 was regarded as the fourth source of uncertainty. No other potential sources of uncertainty (the test force application time, test force duration time, tip radius and length of the line of junction, measuring device (microscope) [12]) were not regarded.

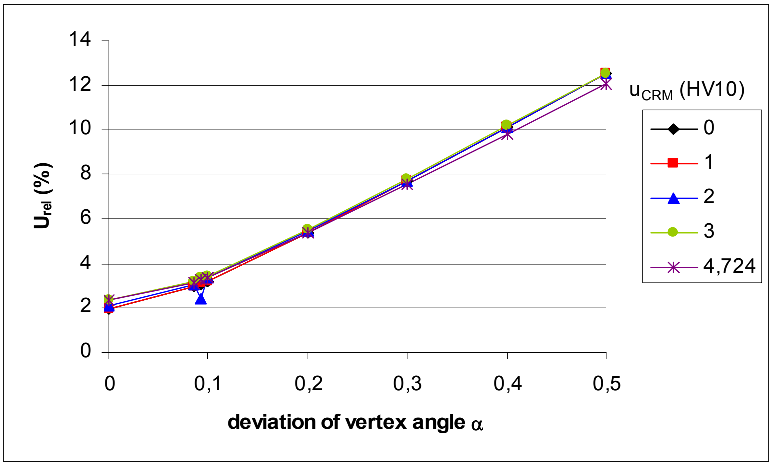

Figure 3 – The influence of vertex angle deviation α and u_{CRM} on mean values of U_{rel} (ΔF = 0.2 %)

The relative maximum permissible error of the tester (relative expanded uncertainty U_{rel}) was calculated using formulas (9) and (11), the coverage coefficient k = 2. The values of U_{rel} for ΔF = -0.2%, α = 0.0925°, individual $\bar{d}_{SD}$ and u_{CRM} = 4.724 HV10 are in tab. 5.

2.3 The discusion

Resulting from tab. 5, all but one results of direct calibrations do not satisfy the requirements of standard. The values of relative expanded uncertainty, calculated according to standard [3] overvalue those obtained by direct calibration. It is result of number with error of calibration E (formulas (2), (8), (10)) in the case of direct calibration. The influence of the vertex angle deviation α on uncertainty is more significant than the influence of test force deviation ΔF, fig. 1 and fig. 2. Increased values of uncertainties were obtained at calibration No. 6, appraiser A. As can be seen on the fig. 3, the influence of u_{CRM} on final U_{rel} is obscure. The higher values of uncertainties in direct calibration published in [14] result from regarded extreme values of test force deviation and first off the vertex angle deviation.

3 CONCLUSION

1. The hardness tester is not conforming for all repeated calibrations using indirect method. Only one calibration (appraiser B) is conform in direct method.
2. The uncertainty obtained by indirect calibration is more significant as it of direct calibration.
3. The influence of the vertex angle deviation on uncertainty is more significant as the influence of test force deviation.
4. With regard to the identified insuficient resolution it is recommended to use larger magnification for the calibration.

ACKNOWLEDGEMENTS

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

REFERENCES

[1] ISO 10 012:2003: Measurement management systems - Requirements for measurement processes and measuring equipment, Available from: http://encarta.msn.com/dictionary_1861594147/calibration.html, Accessed: 2008-07-03.

[2] STN EN ISO 6507-2:2005: Metallic materials. Vickers hardness test. Part 2: Verification and calibration of testing machines.

[3] Miller, C.; Ohno, Y. (2005): Understanding and quantifying uncertainty is key to accurate and cost-effective testing, Available from: http://oemagazine.com/fromTheMagazine/feb05/uncertainty.html, Accessed: 2005-02-28.

[4] ISO/IEC 17 025:2005: General requirements for the competence of testing and calibration laboratories.

[5] STN EN ISO 6507-1:2005: Metallic materials. Vickers hardness test. Part 1: Test method.

[6] Measurement system s analysis (MSA) (2003): Reference manual. Third Edition, pp. 44, 74.

[7] International Vocabulary of Basic and General Terms in Metrology VIM. Available from: http://www.ntmdt.ru/download/vim.pdf , Accessed: 2008-03-14.

[8] Tobolski, E. (2003): Uncertainty in Hardness testing, *Advanced materials & processes*, Vol. 161 No. 5, pp. 25.

[9] Adams, T. M. (2002): G104 - A2LA Guide for Estimation of Measurement Uncertainty In Testing, Available from: http://www.a2la.org/guidance/est_mu_testing.pdf, Accessed: 2007-10-25.

[10] Vickers sensitivity coefficients. Available from: http://resource.npl.co.uk/docs/science_technology/mass_force_pressure/hardness/vickers_hardness_co.pdf Accessed: 2009-10-09.

[11] EA-10/16 EA (2004): Guidelines on the Estimation of Uncertainty in Hardness Measurements.

[12] TPM 0051-1993: Stanovenie neistôt pri meraniach.

[13] Petrík, J. et al. (2008): *The evaluation of the hardness tester quality*, Acta Metallurgica Slovaca, Vol. 14 No. 3, pp. 405 – 413.

ABOUT THE AUTHORS

Doc. Ing. Jozef Petrík, PhD is associated professor in Department of integrated management. He deals with metrology, the first of all quality of metallurgical values measurement.

Adress: Technical University of Košice, Faculty of Metallurgy, Letná 9, 042 00, Košice, Slovakia, tel.: 055 602 2872, e-mail: jozef.petrik@tuke.sk,

Doc. RNDr. Pavol Palfy, PhD is associated professor in Department of integrated management. He deals with quality and environmental management systems.

Adress: Technical University of Košice, Faculty of Metallurgy, Letná 9, 042 00, Košice, Slovakia, tel.: 055 602 2704, e-mail: pavol.palfy@tuke.sk,

ISSN 1335-1745