

LOAD RANGE EXPANSION OF NATIONAL PRIMARY HARDNESS STANDARD MACHINE ON VICKERS SCALES

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Abstract -The current paper describes the results of measurements conducted during the improvement of the national primary hardness standard machine on Vickers scales. As a result of improvement, the range of applied loads has been expanded from 0,098 N to 0,0098 N. This helped to provide the uniformity of hardness measurements on Vickers scales HV0,005, HV0,002 and HV0,001, expand the standard machine application area as well as reduce measurements uncertainty on the primary standard machine as a result of studying geometrical parameters of reference indenters with the help of a new high-precision complex. A base for the development of a new national hardness standard machine in the field of nanoindentation at the loads of less than 0, 0098 N has been created.

Keywords: primary hardness standard machine, Vickers scale HV0,001, measurements uncertainty.

1. INTRODUCTION

In 2006 the improved national primary hardness standard machine on Vickers scales GET 31-06 was accepted with an expanded Vickers measurement range up to micro-hardness scales. However, in GET 31-06 the hardness measurement range was limited by the lowest test load of 0,098 N and length of the restored indentation diagonal of 20 μm [1]. The development of new technologies and the apparition of a wide stock of micro hardness testers with test loads of 0,0098 N and higher, that haven't been introduced into the current verification scheme, have set the problem of providing the uniformity of Vickers hardness measurements in the load range between 0,098 N and 0,0098 N. In 2009-2010 the works on further improvement of the standard machine were conducted. The improved standard machine has been approved and assigned the registration number GET 31-10.

2. STANDARD MACHINE METROLOGICAL CHARACTERISTICS

The national primary hardness standard machine on Vickers scales provides the reproduction of hardness numbers on Vickers scales (HV) in the range of 8- 2000 with an error characterized by the repeatability of hardness numbers from 1 HV to 80 HV and the systematic error from 1 HV to 60 HV depending on the load and the nominal value of the hardness number. As a result of studies, the mathematical model of uncertainty calculation was specified and the assessment of hardness measurement result uncertainty for all Vickers hardness scales was carried out. For each scale the calculation of expanded uncertainty was carried out on the basis of a mathematical model, and then the interpolation equation on the basis of a second-degree polynomial was calculated. Table 1 shows the expanded uncertainty of measurement results on the standard machine. The uncertainty data are specified for the coverage factor 2 at the confidence level of 95% .

Table 1. Expanded uncertainty of measurement results on the GET 31-10.

Scale	Load	Expanded uncertainty
HV0,001	9,8 mN	$0,084 \times \text{HV} + 5,1\text{E-}5 \times \text{HV}^2$
HV0,002	19,6 mN	$0,08 \times \text{HV} + 3,0\text{E-}5 \times \text{HV}^2$

HV0,005	49,0 mN	$0,062 \times \mathbf{HV} + 3,5\mathbf{E-5} \times \mathbf{HV}^2$
HV0,01	98,1 mN	$0,040 \times \mathbf{HV} + 4,3\mathbf{E-5} \times \mathbf{HV}^2$
HV0,025	245,2 mN	$0,049 \times \mathbf{HV} + 3,5\mathbf{E-5} \times \mathbf{HV}^2$
HV0,05	490,3 mN	$0,063 \times \mathbf{HV} + 4,2\mathbf{E-5} \times \mathbf{HV}^2$
HV0,1	980,7 mN	$0,004 \times \mathbf{HV} + 3\mathbf{E-5} \times \mathbf{HV}^2$
HV0,2	1,96 N	$0,029 \times \mathbf{HV} + 2\mathbf{E-5} \times \mathbf{HV}^2$
HV0,3	2,94 N	$0,024 \times \mathbf{HV} + 2\mathbf{E-5} \times \mathbf{HV}^2 + 0,2$
HV0,5	4,9 N	$0,02 \times \mathbf{HV} + 1,5\mathbf{E-5} \times \mathbf{HV}^2$
HV1	9,8 N	$0,0011 \times \mathbf{HV} + 1\mathbf{E-5} \times \mathbf{HV}^2 + 0,7$
HV2	19,6 N	$0,0092 \times \mathbf{HV} + 7\mathbf{E-6} \times \mathbf{HV}^2 + 0,1$
HV3	29,4 N	$0,0075 \times \mathbf{HV} + 6\mathbf{E-6} \times \mathbf{HV}^2$
HV5	49,0 N	$0,0048 \times \mathbf{HV} + 6\mathbf{E-6} \times \mathbf{HV}^2$
HV10	98,1 N	$0,0039 \times \mathbf{HV} + 3\mathbf{E-6} \times \mathbf{HV}^2 + 0,4$
HV20	196 N	$0,0071 \times \mathbf{HV} + 5\mathbf{E-6} \times \mathbf{HV}^2 + 0,3$
HV30	294 N	$0,0075 \times \mathbf{HV} + 2\mathbf{E-6} \times \mathbf{HV}^2 + 0,6$
HV50	490 N	$0,0048 \times \mathbf{HV} + 3\mathbf{E-6} \times \mathbf{HV}^2 + 0,2$
HV100	980 N	$0,0035 \times \mathbf{HV} + 2\mathbf{E-6} \times \mathbf{HV}^2 + 0,3$

The standard machine metrological characteristics are applied for indentations with the diagonal size exceeding 2 μm .

3. STANDARD MACHINE DESCRIPTION AND COMPOSITION

The measuring instruments comprising of which is the primary standard machine are shown in p. [2]. For the aims of improvement, a nano hardness tester and a new high-precision complex for the measurement indenters' geometrical parameters were introduced into the standard machine composition. Indentation according to the procedure corresponding to Vickers scales at the loads of 9,8 mN, 19,6 mN and 49,0 mN was conducted on the nano hardness tester, while the measurement of the diagonals' length was carried out on an atomic-force microscope.

4. RESULTS OF STANDARD MACHINE STUDY

The main contribution to hardness measurements uncertainty on Vickers scales is made by the values shown in Table 2.

Table 2. Results of the study of quantities contributing to measurements uncertainty on GET 31-10 on Vickers scales.

Measured values X_i	Symbol	Unit	Nominal value		Maximum deviation during measurements, \pm	
Load	F	N	0,00981	9,81	8×10^{-8}	$7,5 \times 10^{-3}$
			0,0196	19,6	1×10^{-7}	$7,5 \times 10^{-3}$
			0,049	29,4	1×10^{-7}	$9,8 \times 10^{-3}$
			0,0981	49,0	3×10^{-5}	$9,8 \times 10^{-3}$
			0,49	98,1	5×10^{-4}	$1,5 \times 10^{-2}$
			0,981	294,3	1×10^{-3}	$1,5 \times 10^{-2}$
			1,96	490,3	5×10^{-3}	2×10^{-2}
			4,9	980,7	$7,5 \times 10^{-3}$	3×10^{-2}

Diagonal length	D	mm	0,002...0,02 0,02...0,08 0,08...0,3 0,3...0,5 0,5...1,0	6×10^{-5} 3×10^{-4} 4×10^{-4} 6×10^{-4} 1×10^{-3}
Apex angle α	α	°	136°	0,02 0,2 0,2 0,02
Tip radius	R	mm	0	3×10^{-4} 3×10^{-4} 2×10^{-4} 1×10^{-4}
Length of line of junction	c	mm	0	5×10^{-4} 5×10^{-4} 4×10^{-4} $1,5 \times 10^{-5}$

The table shows geometrical parameters for four reference indenters.

4.1. Uncertainty of load measurement results

Direct calibration of the test loads from 0,009807 N to 0,09807 N was conducted with the help of SE2 microbalance. At that the tests were carried out in three independent series. The results of the calibration are demonstrated in Table 3.

Table 3. Calibration protocol of test load 0,009807 N on G200 nano hardness tester.

Calibrated load value F_c , mN	Calibration results			Mean value F , mN	Root mean square deviation of the three load measurement series, %
	1. series F_1 , mN	2. series F_2 , mN	3. series F_3 , mN		
9,8158	9,8151	9,8152	9,8152	9,8149	0,003
	9,8144	9,8144	9,8146		
	9,8151	9,8149	9,8143		
	9,8151	9,8149	9,8145		
	9,8152	9,8149	9,8149		
Mean value in a series	9,8150	9,8149	9,8147		
deviation $F_c - \bar{F}_i$, mN	-0,0008	-0,0009	-0,0011		

The temperature drift during the tests did not exceed 1 °C, humidity fluctuated from 45 % to 50 %. SE2 balance relative error during the measurement of a 0,001 kg mass

equals $8 \cdot 10^{-4}$ % according to the verification certificate. The measurement result relative uncertainty due to the temperature drift does not exceed $2 \cdot 10^{-3}$ %. The combined relative uncertainty of the measurements by the load equals $3,8 \cdot 10^{-3}$ %.

4.2. Uncertainty of the indentation diagonal length measurement results

The maximum acceptable errors of measuring devices with the help of which the indentation diagonal lengths are measured, are specified in p. [1]. A detailed description of an automated optical subsystem of the primary standard machine for the measurements of indentations' diagonal lengths of 20 μm and more as well as the results of its study are listed in [2]. The measurement of indentations' diagonal lengths of less than 20 μm was conducted with the help of an atomic-force microscope. The microscope calibration was carried out using a fragment of a diffraction grating that had been preliminarily calibrated using a helium-neon laser with the wavelength of 632,8 μm . The relative expanded uncertainty of the measurements result does not exceed 3 %.

4.3. Uncertainty of measurements of diamond indenters' parameters

The results of measurements of Vickers diamond indenters' parameters are shown in Table 4 and were carried out with the help of the high-precision metrological complex [3]. The complex helps to measure all geometrical parameters of Vickers diamond indenters with the uncertainty of: the angle between the faces at the vertex of $0,1^\circ$, flatness of 0,0003 mm, linearity of 0,001 mm, the line of junction of 0,00025 mm.

Table 4. Results of measuring reference diamond indenters' parameters.

Parameter	Measurement result	Expanded uncertainty
Indenter No 1		
Mean angle between faces	135,98°	0,08°
Length of line of junction	0,5 μm	0,5 μm
Flatness deviation	2,75 μm	0,27 μm
Indenter No 2		
Mean angle between faces	136,32°	0,17°
Length of line of junction	0,5 μm	0,5 μm
Flatness deviation	2,75 μm	0,27 μm
Indenter No 3		
Mean angle between faces	135,83°	0,1°
Length of line of junction	0,5 μm	0,2 μm
Flatness deviation	1,9 μm	0,27 μm
Indenter No 4		
Mean angle between faces	136,02°	0,08°
Length of line of junction	0,15 μm	0,1 μm
Flatness deviation	1,5 μm	0,14 μm

The obtained metrological characteristics of indenters have helped to improve the characteristics of GET 31-06 primary standard machine.

4.4. Reference hardness test blocks study

Reference hardness test blocks were used for the standard machine study. Table 5 shows the results of comparative tests on hardness test blocks.

Table 5. Comparative results of reference test blocks measurements

№	Test block type	Yamamoto scientific tool laboratory		VNIIFTRI	
		Hardness number, HV	Repeatability, HV	Hardness number, HV	Repeatability, HV
1	HN-W430 (Single crystal tungsten)	391 HV 0,01	7	410 HV 0,01	8
		405 HV 0,001	16	412 HV 0,001	12
2	UMV200 (SK85, C1720P)	200 HV 0,01	8	210 HV 0,01	7
		198 HV 0,002	5	210 HV 0,005	10
				207 HV 0,002	5
				213 HV 0,001	12
3	UMV500 (SK85, C1720P)	505 HV 0,01	15	516 HV 0,01	11
		500 HV 0,002	19	520 HV 0,005	13
				524 HV 0,002	12
				524 HV 0,001	10
4	UMV700 (SK85)	699 HV 0,01	8	709 HV 0,01	13
		699 HV 0,002	10	712 HV 0,005	16
				709 HV 0,002	14
				705 HV 0,001	14
5	UMV900 (SK85)	905 HV 0,01	9	921 HV 0,01	20
		906 HV 0,002	10	920 HV 0,005	22
				915 HV 0,002	16
				918 HV 0,001	17
6	HMV30 (Au)	34.2 HV 0,01	2,7	33,5 HV 0,01	0,9
		35.0 HV 0,001	2,4	34,7 HV 0,001	1,1

The comparison of the results Yamamoto scientific tool laboratory and VNIIFTRI has shown that hardness test blocks can be used for the transfer of HV0,001; HV0,002; and HV0,005 hardness scales to industrial measuring instruments.

4.5. The study of expanded measurement uncertainty on the standard machine

Table 6 shows the scheme of calculating expanded uncertainty of hardness measurements' result and corrections that was used during BIPM key international comparisons on Vickers scales [4, 5].

Table 6. Scheme of correction calculation and estimation of hardness measurement expanded uncertainty while making measurements on test block 213 HV0,001.

Measured quantity X_i	Symbol	Unit	Value	σ_{x_i}	s_i	a_i	$u^2(x_i)$	c_i	σ_{ω}	$u^2(y_i)$
Load F	F	N	0,01	0,000	3,80E-07		1,4E-13	2,2E+04	0,0E+00	6,8E-05
Indentation diagonal	d	mm	0,003	0		0,00006	1,2E-09	-1,4E+05	0,0E+00	2,5E+01
Apex angle a	$^\circ$	$^\circ$	136	-8,7E-04	0,00175		3,0E-06	4,3E+01	-3,8E-02	5,7E-03
Tip radius Dr	r	mm		1,5E-04		1,0E-04	3,3E-09	-8,4E+04	-1,3E+01	2,3E+01
Length of line of junction Dc	c	mm		2,0E-04		1,0E-04	3,3E-09	1,0E+05	2,0E+01	3,4E+01
Total									7,65	8,3E+01

Table 3 shows that the standard uncertainty of the reference test block with the nominal value of 213 HV equals $u = 9,1$ HV, while the expanded uncertainty equals 18,3 HV, for the coverage factor $\kappa = 2$ at the confidence level of 95%.

5. CONCLUSION

The improvement of the national primary standard machine has helped to ensure the uniformity of hardness measurements on Vickers scale in the load range from 98,1 mN to 9,8 mN. The following results have been obtained:

- The standard machine reproduces hardness scales at the loads of 9,8 mN, 19,6 mN and 49,0 mN. The error of the test load reproduction is less than 1 %.
- The minimum indentation diagonal length equals 2 μm . The uncertainty of the diagonal length measurement does not exceed 3 %.
- The uncertainty of indenters' parameters measurements while measuring their parameters on the high-precision complex has been reduced.
- Micro hardness test blocks for the loads of 9,8 mN, 19,6 mN and 49,0 mN can be used for the transfer of Vickers scale in this range.
- A base for the creation of the national hardness standard machine in the field of nanoindentation has been prepared.

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