XIX IMEKO World Congress
Fundamental and Applied Metrology
September 6–11, 2009, Lisbon, Portugal

INSTALLATION AND UNCERTAINTY EVALUATION OF REFERENCE HARDNESS STANDARD OF CROATIA

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Abstract – Reference hardness standard with HV1 – HV50 measuring range was installed at LIMS (Laboratory for Testing Mechanical Properties) at the end of 2007 with the aim of providing continuous improvement of the metrology infrastructure of Croatia. The evaluation of the measurement uncertainty is one of the most important tasks for establishing this reference standard. Therefore, the influence quantities contributing to the uncertainty were determined and the machine calibrated by direct and indirect method. In this paper the results of the measurements are presented and discussed.

Keywords: hardness of metals, reference hardness standard, measurement uncertainty

1. SCOPE

A reference hardness machine has been provided to represent the hardness reference standard in Croatia. The machine was made by Indentec, England, type 5030TKV Std. with hardness scale for Vickers. Vickers measurement in the range of force from 9,807 N up to 490,3 N should be realized with uncertainties which assure accurate measurement in the Croatian industry.

This reference hardness machine is intended to:

- satisfy the needs of the Croatian industry for arbitrary measurement,
- raise the calibration service to a higher level.

2. INSTALLATION

Hardness reference machine 5030TKV (Fig. 1) was installed in the Laboratory for Testing Mechanical Properties at the Faculty of Mechanical Engineering and Naval Architecture. The load is realised by means of weights and a lever. By definition, force is defined with the expression $F=m\cdot g$. Therefore, the gravimetric measurements on the location of the reference hardness machine installation were needed in order to achieve the appropriate load by means of weights. Relative gravimeter CG-3M AutoGrav produced by Scintrex, Canada was applied for the gravimetric measurements. This is a quartz, microprocessor-controlled and highly automated instrument. The working scope of the gravimeter is over 7000 mGal (1 mGal = 10^{-5} ms^{-2}), which encompasses the region of the entire Earth, not needing resetting, and the standard resolution of the gravimeter is 1 µGal (1 µGal = 10^{-8} ms^{-2}). Measuring by means of gravimeter Scintrex CG-5 is done automatically eliminating thus the measurer's assessment error, and the measurement data are stored in the gravimeter memory. The acceleration of gravitation g = 98066333,7 $*10^{-08}$ ms^{-2} ± 4,7$*10^{-08}$ ms^{-2} was obtained by analysis and calculation. On the basis of that value the weights were produced.

Figure 1. Reference hardness machine 5030TKV

3. UNCERTAINTY CALCULATION

At first, the main sources of measurement uncertainties are determined and then they are evaluated by measurement. The main parameters which have an effect on measurement uncertainty are those defining the measurement process. They are stipulated and given by the standard EN ISO 6507. The estimation and calculation of the measurement uncertainty was carried out in accordance with the EN ISO 6507-2 [1], EN ISO 6507-3 [2] and document EURAMET/cg-16/v.01[3].

3.1 Test force

The applied force was calibrated with force transfer standard (load cells). Such a system leads to two main error sources:

- measurement of the applied force;
- measurement of the maintenance of the applied force.

Several series of measurements were carried out in accordance with the recognized standards. The maintenance of the applied force was assessed by analysing the data obtained during force calibration.

Loading forces were checked by the portable standard of force of class 00 in accordance with EN ISO 376 standard. They were calibrated at PTB (Pysikalisch-Technische Bundesanstalt), Germany, using national standards of force (deadweight machines) of measuring capability ≤ 0,002 %. The expanded measurement uncertainty of their calibration was ≤ 0,03 %. For each loading two different portable standards of force were applied.

The uncertainty of measurement of the test force is evaluated in two steps:

- determination of the combined relative standard uncertainty of the reference value of the force transfer standard:

$$u_{FRS} = \sqrt{u_{TS}^2 + u_T^2 + u_S^2 + u_D^2} \tag{1}$$

u_{TS} - relative uncertainty of the transfer standard
u_T - relative uncertainty due to the temperature deviation
u_S - relative long term stability,
u_D - relative interpolation deviation.

- determination of the measurement uncertainty of each force.

The uncertainty of measurement of the test force is calculated by using the following formula:

$$u_F = \sqrt{u_{FRS}^2 + u_{FHTM}^2 + u_{main}^2} \tag{2}$$

u_{FHTM} – uncertainty of measurement of the test force – normal distribution
u_{main} – uncertainty of maintenance of the test force – rectangular distribution

Expanded uncertainty:

$$U_F = k \times u_F \tag{3}$$

Each force was measured three times in three different indentation positions. Results are shown in Table 1.

Table 1: Results of force measurement uncertainty

F, N	F_{sr}, N	ΔF_{rel}, %	U_F, %
9,807	9,817	0,102	0,061
29,42	29,431	0,038	0,049
49,03	49,049	0,039	0,067
196,1	196,198	0,050	0,052
294,2	294,410	0,071	0,053
490,3	490,350	0,010	0,037

F_{sr} - mean value of the measured forces,
ΔF_{rel} - relative deviation of the test force,
U_F - relative expanded uncertainty of the test force; k=2.

3.2 Test length

Vickers indentations are measured using the optical length measuring system. The opto-electric system CCD-LFSB consists of an optic microscope with the high precision CCD camera which is connected to the computer and appropriate software (Fig. 2).

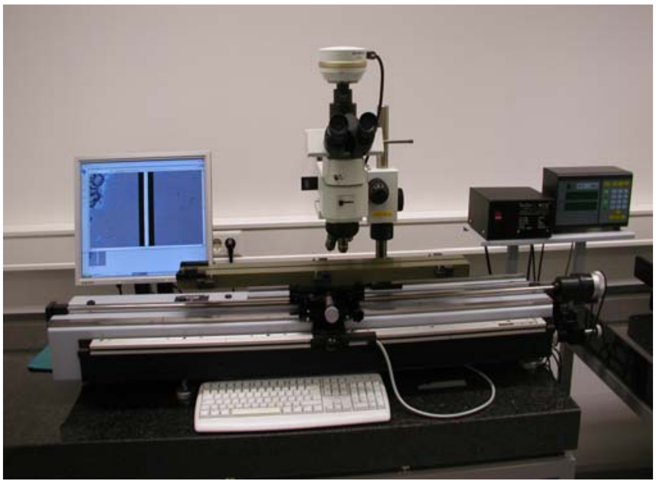

Figure 2. Opto-electric system CCD-LFSB for length measurements

The process of image analysis is static which means that the images need to be processed. It is not possible to process "live" image from the CCD camera. The CCD-LFSB system was calibrated by the reference measuring scale (transfer standard) which was calibrated at PTB (Physikalisch-Technische Bundesanstalt), Germany, and the measurement uncertainty is given by the expression:

$$U_{LRS} = \sqrt{(10nm + (0{,}18 \bullet L)^2} \tag{4}$$

with k=2 and P=95 % where U_{LRS} is expressed in nm, and L in mm. The uncertainty of length measurement is calculated as follows:

$$u_L = \sqrt{u_{LRS}^2 + u_{ms}^2 + u_{LHTM}^2} \tag{5}$$

u_{LRS} – relative uncertainty of the transfer standard,
u_{ms} – relative uncertainty due to the resolution,
u_{LHTM} – relative standard uncertainty of measurement results.

Expanded uncertainty:

$$U_{\mathrm{L}} = k \times u_{\mathrm{L}} \tag{6}$$

During preliminary measurements, an error was noted due to the alignment of print edges (Fig. 3).

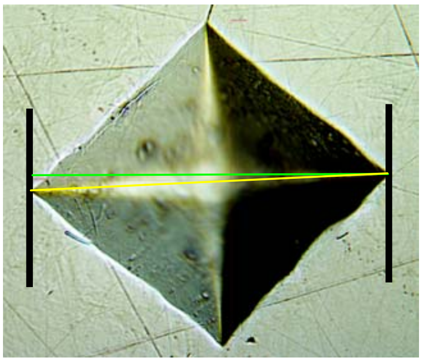

Figure 3. Error due to print rotation

Based on the research carried out at NPL, and presented in literature [4] and observation of the CCD - LFSB system behaviour, this error was assessed and included in the estimate and calculation of the measurement uncertainty. Table 2 shows the results of checking the optical measurement CCD-LFSB system.

Table 2: Results of length measurement uncertainty

Length, mm	ΔL_{rel}, %	U_{L} %
0,05 – 0,8	0,11	0,15

ΔL_{rel} - maximum relative deviation of length,
U_{L} - maximum expanded uncertainty of length; k=2,

3.3 Indenter

The indenter was bought as a high class indenter and calibrated at Star Industrial Tools Ltd., England.

Table 3: Results of diamond indenter verification

Measurement	Requirement	Result	Uncertainty of measurement
Angles in a section normal to the axis of the diamond pyramid	A1–B1 90°±0.4°	89°98′	5 mins of arc
	B1–C1 90°±0.4°	90°00′	
	C1–D1 90°±0.4°	90°01′	
	D1–A1 90°±0.4°	90°01′	
Inclination of the axis of the pyramid to the axis of the seating surface	A ±0,30°	68°03′	5 mins of arc
	B ±0,30°	68°02′	
	C ±0,30°	68°00′	
	D ±0,30°	68°00′	
Angle between opposite faces of the diamond pyramid	A-C 136° ± 0,1°	136°03′	5 mins of arc
	B-D 136° ± 0,1°	136°02′	
Line of junction between opposite faces	Normal 0,001mm	0,92µm	0,5µm
	Micro 0,00025mm		

The numerical results are shown against the values specified in EN ISO 6507-3.

3.4 Test cycles

All the phases of the test cycle are adjustable at the machine panel and they are within tolerances required by the standards. Test time is calibrated by means of a stopwatch.

3.5 Uncertainty calculation for indirect calibration

Indirect calibration is carried out using high quality reference hardness blocks made by Indentec, England. The uncertainty of measurement of indirect verification of the reference hardness testing machine is calculated by the equation:

$$u_{\mathrm{CM}} = \sqrt{u^2_{\mathrm{CRM\text{-}P}} + u^2_{\mathrm{xCRM-1}} + u^2_{\mathrm{CRM-D}} + u^2_{\mathrm{ms}}} \tag{7}$$

$u_{\mathrm{CRM\text{-}P}}$ - calibration uncertainty of the reference block without its inhomogeneity according to the calibration certificate for k=1,
$u_{\mathrm{xCRM\text{-}1}}$ - standard uncertainty due to the repeatability of the reference hardness machine,
$u_{\mathrm{CRM\text{-}D}}$ - long time drift in hardness of the reference block,
u_{ms} - relative uncertainty due to the resolution.

$$u_{\mathrm{xCRM\text{-}1}} = \frac{t \times s_{\mathrm{xCRM\_1}}}{\sqrt{n}} \tag{8}$$

$s_{\mathrm{xCRM\text{-}1}}$ - standard deviation due to the repeatability,
t = 1.15 - student factor,
n = 5 …- number of indentations.

Expanded uncertainty:

$$U_{CM} = k \times u_{CM} \tag{9}$$

The calibration results for HV10 and HV30 are presented in Table 4 whereas the calibration results for HV1; HV3; HV5; HV20 and HV50 are not presented in this paper because the calibrations were carried out only with a single primary standard plate for each loading.

Table 4: Results of indirect calibration for HV10 and HV30

	Hardness value	$\overline{HV}$ HV	U_{HTM} HV (k=2)	$\lvert\overline{b}\rvert$ HV	$\Delta H_{\mathrm{HTMmax}}$ HV
HV10	272,2 HV10	273,10	1,66	0,90	2,56
	457,4 HV10	45632	2,97	1,08	4,05
	807,9HV10	806,17	5,65	1,73	7,39
HV30	275,0 HV10	274,52	1,76	0,48	2,24
	470,7 HV10	469,20	3,03	1,50	4,53
	802,0 HV10	800,07	5,20	1,93	7,13

3.6 Intercomparison measurements

Intercomparison measurements are underway and they are being carried out with two other national laboratories with the published CMCs. The results of these measurements will be published at one of the future metrology symposia.

4. CONCLUSION

The results of direct calibration have shown that all the requirements were fulfilled according to EN ISO 6507-3. The test force for the method HV 1 was on the edge of the permitted deviations. This can be also attributed to the transfer standard uncertainty for the given force. The uncertainty contribution due to the repeatability of the reference hardness machine is small. The uncertainty contribution due to the inhomogeniety of the blocks u_{xCRM} is also small as the result of high quality blocks which were used for measurement. On the basis of indirect calibration results (for HV 10 and HV 30) it can be concluded that the expanded measurement uncertainty of the reference hardness machine will be in the range ±1 % HV for the entire measuring range of the machine. This will surely satisfy the needs of the Croatian metrology infrastructure.

REFERENCES

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