Uncertainty calculations and calibration of metal hardness testing equipment

S. Kaan Gürel[1,*], *İ. Ahmet* Yüksel[1] , *T .Oytun* Kılınç[1]

[1]Department of Laboratories and Calibration Management, Roketsan Missiles Industries Inc., 06780 Ankara, Turkey

Abstract. Hardness tests have vital role for industries such as aerospace, ground vehicles and energy. In these industries, companies do not have only their internal processes but also sub-contractors. Therefore, for acceptance tests, hardness testing becomes so convenient way in order to confirm conformance of incoming materials to the facility. Additionally, there are also critical test limits that comes from technology transfer projects. In these type of projects, specification limits can be very narrow and design of parts may not be changed and quality (non-conformance) decisions may not be taken as rework or used as. In case of probable failure, material parts could cause catastrophic results in mentioned industries. In this study, hardness test capability of a Vickers Hardness Machine is investigated by use of direct and indirect calibration methods. Uncertainty calculations are made for Vickers test system. While making calibration of hardness test machines, the parts which are components of hardness scales shall be also calibrated / verified. These components must be in necessary tolerances related to EN/ISO specifications. After that, calibration of these hardness test machines are made by use of hardness reference blocks that are produced and calibrated by national metrology institute or an accredited laboratory.

1. Introduction

Hardness is defined as the resistance of a material to an indentation. Hardness gives significant clues about different properties of materials such as tensile strength, heat treatment condition, resistance to wear, abrasion, scratch etc. In order to obtain an idea about these properties, the most convenient test is hardness test because it is one of the most simple and practical test. Additionally it may be accepted as non-destructive test for many application and gives mechanical outputs. There are many hardness tests currently in use for a great range of materials from soft rubber to hard ceramics. In this study, tests are conducted via metallic materials. Principle of hardness test depends on indentation amount of indenter and dimension of marks left by indenter on the test specimen. Accordingly, calibration, verification and uncertainty calculations are investigated in this study for a Vickers hardness test machine (Emcotest Durascan G5).

* Corresponding author: kaan.gurel@roketsan.com.tr

2. Methodology

EN ISO 6507 is used as reference test specification. All tests, calibration processes and uncertainty measurements are done according to this standard. Vickers method is widely used for different metals having wide hardness range. In this method, a diamond is used as indenter in the form of a right pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a load of 1 to 100 kgf (see Fig. 1). The full load is normally applied for 10 to 15 seconds [1]. Two diagonals of test marks are measured by use of an optical system. Calculated average diagonal value is inserted in hardness equation which provides obtaining the hardness value of the material. The Vickers hardness is the quotient obtained by dividing the kgf load by the square mm area of indentation.

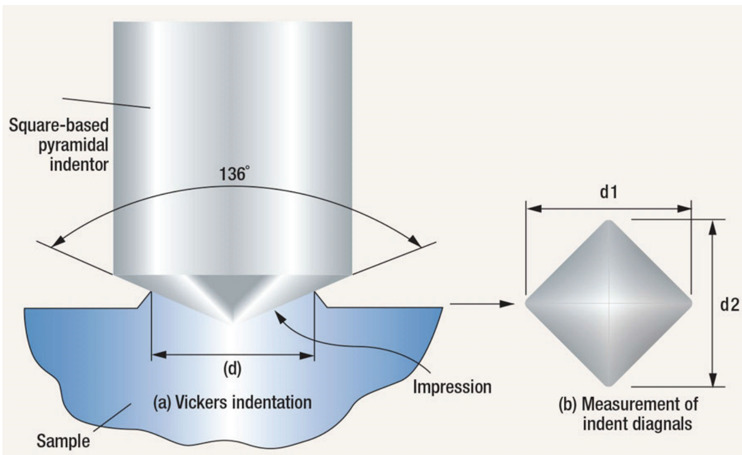

Fig. 1. Illustration of diamond indenter and test mark [1].

$$Vickers\ Hardness\ HV = \text{Constant} * \frac{Test\ Force\ (F)}{Area\ of\ Test\ Mark\ (A)} \quad (1)$$

$$HV = 0.102 * \frac{2F * \text{Sin}\frac{136°}{2}}{d^2} = 0.189 * \frac{F}{d^2} \quad (2)$$

d: Average of d_1 and d_2

2.1 Direct calibration of Vickers test method

There are four main steps in direct calibration method.

2.1.1 Calibration of force application system

One of the main components of a hardness measurement is force application system. Because of this system is calibrated by use of referenced load cell. Load cell shall be placed into test table so that test force is applied to the load cell. Load cell shall be at least class 1 according to ISO 376 [2]. There shall be taken three measurements for every desired test load. Any exception in tolerances cannot be accepted during these measurements. There is a table below showing the necessary tolerances.

Table 1. Test force range against tolerances.

Test Load Range	Tolerances
1,961 F ≤	± 1,0 %
0,09807 ≤ F < 1,961	± 1,5 %
0,009807 ≤ F < 0,09807	± 2 %

2.1.2 Verification of indenter

Secondly, Indenter geometry shall be verified. Primarily, a visual inspection is recommended on indenter to detect any contamination or defect. Indenter must have a calibration certificate provided by an accredited laboratory. Statements shown below must be verified in the certificate:
- The four faces of the square-based diamond pyramid shall be polished and free from surface defects
- The verification of the shape of the indenter can be made by direct measurement or optical measurement. The device used for the verification shall have a maximum expanded uncertainty of 0.07°.
-The measured angles between the opposite faces at the vertex of the diamond pyramid shall be within the range 136° ± 0.5° (see Fig.1)
-The angles between the opposite faces also be verified by measuring the angle between the opposite edges. To meet requirements, the angles between the opposite edges shall be 148,11° ± 0.76°
-The angle between the axis of the diamond pyramid and the axis of the indenter-holder (normal to the seating surface) shall be less than 0.5°
-The four faces should ideally meet at a common point, however, there is usually a line of junction, a, between opposite faces as shown in figure 2. The length of the line of junction shall be determined by direct measuring the indenter tip or by measuring the tip impression in an indentation.

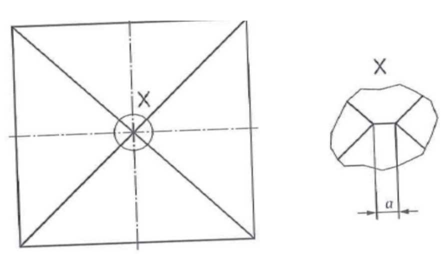

Fig. 2. Line of junction on the top of the indenter [2].

The maximum permissible length of the line of junction between opposite faces is given in Table 2.

Table 2. Line of Junction Tolerance

F Test Force Range, N	Max. Permissible length of the line of junction, a , mm
F ≥ 49,03	0,002
1,961 ≤ F < 1,961	0,001
0,09807 ≤ F < 1,961	0,0005

2.1.3 Verification diagonal measuring system

In hardness measurement systems, diagonal measuring system has a vital role. Proper measurement of test mark is critical as generation of test mark because hardness value is given just after measurement of diagonals in the end. In last decades, microscope of hardness test machines and proper software are communicated with each other and system decides to necessary objective in order to measure diagonals. Therefore, all objectives shall be verified for certain hardness ranges by a calibrated stage micrometer. At least five measurements are taken. Each of them shall consist of at least three measurement series. The maximum permissible error of each of three measurements at each interval shall be as indicated in Table 3.

Table 3. Calibration and verification requirements of the diagonal measuring system [2].

Measurement Parameters	Calibration and Verification Requirements
Max. expended uncertainty of the distances between the line intervals on the stage micrometer	Greater of 0.0004mm or 0.2%
Max. permissible error of the measurements of the stage micrometer intervals	Greater of 0.0008mm or 1%

2.1.4 Verification of test cycle

The last step that shall be checked is test cycle. This measurement is taken while the test force is applied. A chronometer having an extended uncertainty of 1 second can be used for this measurement.

Test force shall be reached between 2-8 seconds. Low load or micro-hardness tests application rate shall not exceed 0.2 mm/seconds. Passed time in force application shall be 10-15 seconds from initial force [2].

2.2 Indirect calibration of Vickers test method

After calibration of constituents of test system, all system is calibrated by use of referenced hardness blocks having a certificate acc. to ISO 6507-3. If machine wanted to be calibrated for only one force, three reference blocks shall be used. However, when verifying more than one test force, at least two reference blocks shall be selected from the hardness ranges specified below for each test force that machine will be verified [3].

- < 250 HV
- 400 HV to 600 HV
- > 700 HV

Five test marks shall be generated on each test blocks. For ease of calculations, it is stated by step by step.

- d1, d2, d3, d4 and d5 are named as mean diagonal of 5 marks
- Arrange diagonal values in increasing order from d1 to d5
- Generate hardness via diagonal measurement
- Arrange hardness values in increasing order from H1 to H5
- Calculate mean diagonal value, d_{mean}= (d1+d2+d3+d4+d5) /5
- Calculate mean hardness value, H_{mean}= (H1+H2+H3+H4+H5) /5
- Note hardness of reference block, H_R

2.2.1 Repeatability

If result of d5-d1is less or equal to 0.0001 mm, repeatability performance of test system is satisfactory. In the contrary case, repeatability shall be evaluated according to Table 4.

$$r_r = 100 * (H5 - H1)/Hmean \quad (3)$$

r_r : Percentage repeatability

Table 4. Maximum permissible relative repeatability.

Vickers Hardness of Reference Block	Max. Permissible Relative HV repeatability of the testing machine, r %HV		
	HV5 to HV100	HV0.2 to <HV5	<HV0.2
HV≤250	6	12	18
HV>250	4	8	12

2.2.2 Bias

Percentage bias calculation is shown in Eq. 4.

$$b_p = 100 * (\mathrm{Hmean} - \mathrm{Hr})/Hr \quad (4)$$

Maximum permissible bias is shown in Table 5.

Table 5. Maximum permissible percentage bias

Mean diagonal length d (mm)	**Max. permissible b_p of testing machine ± %HV**
$0.02 \leq d_{mean} < 0.14$	$0.21/d_{mean}+1.5$
$0.14 \leq d_{mean} < 1.4$	3

3. Application and measurements

In this study, both direct and indirect verifications are done. However, it is important to indicate that uncertainty calculations are divided in two different ways. One of them depends on direct verification, while other one depends on indirect verification. Considering literature for industry, to make uncertainty calculation based on indirect verification method keeps us more safety side for test system [4].

Uncertainty calculations based on direct verification methods include force application system, diagonal measurement system, indenter geometry and test cycle. If there is an unpredictable problem on hardness test equipment such as geometrical damage on seat of test area, it is meant that uncertainty calculation is missing an important parameter that affects test result. Therefore, it can be seen that indirect uncertainty calculation eliminates unexpected errors.

Uncertainty calculations based on indirect verification directly give output about hardness values instead of its sub-constituents such as force application or indenter geometry. This uncertainty budget only consists of constituents about reference hardness block and diagonal measuring system. Diagonal measuring system appears in both uncertainty budget list because it directly affects hardness value of materials.

3.1 Measurement on force application system

Twelve measurements are taken in order to verify four different test type by use of a reference load cell. Test result can be observed in Table 6.

Table 6. Force application system verification measurements

Type	Reference Force (N)	Measured Force (N)	Max. Permissible Bias (%)	Bias (%)	Repeatability (%)	Measurement Uncertainty (%)
HV0.1	0.9807	0.99	1	0.91	0.3	0.16
		0.99		0.70		
		0.99		0.90		
HV1	9.807	9.90	1	0.69	0.16	0.16
		9.88		0.89		
		9.90		0.59		
HV5	49.035	49.3	1	0.53	0.11	0.16
		49.29		0.51		
		49.34		0.63		
HV10	98.07	98.15	1	0.08	0.01	0.16
		98.15		0.08		
		98.15		0.08		

According to results above, force application system of Vickers hardness machine is satisfactory.

3.2 Measurement on indenter

According to requirements of 2.1.2, indenter is completely checked on manufacturer's certificate.

Table 7. Diamond indenter verification measurements.

		Expected Value	Measured Value	Bias	Tolerance	Uncertainty
Face angles		136°	135.797°	-0.203°	±0.5°	0.040°
Axial Deviation				0.07°	±0.5°	0.12°
Offset	≥ HV0.01 ≥ HV0.2 ≥ HV0.5	Acc. To Table 2	0.00027mm		<0.0005mm <0.0010mm <0.0020mm	0.038µ

3.3 Measurement on diagonal measuring system

Fifteen measurements are taken by a reference stage micrometer in order to verify five different series. Test results are shown in Table 8.

Table 8. Verification of diagonal measurement system.

Reading from Reference (µm)	Measurements from Measuring System (µm)	Bias (%) or µm	Max. Permissible Bias (%) or µm	Measurement Uncertainty (µm)	Max. Permissible Measurement Uncertainty (µm)
20	20.23	0.23 µm	0.8 µm	0.1 µm	0.4 µm
20	20.23	0.23 µm	0.8 µm	0.1 µm	0.4 µm
20	20.23	0.23 µm	0.8 µm	0.1 µm	0.4 µm
40	40.07	0.07 µm	0.8 µm	0.1 µm	0.4 µm
40	40.07	0.07 µm	0.8 µm	0.1 µm	0.4 µm
40	40.07	0.07 µm	0.8 µm	0.1 µm	0.4 µm
60	60.17	0.17 µm	0.8 µm	0.1 µm	0.4 µm
60	60.17	0.17 µm	0.8 µm	0.1 µm	0.4 µm
60	60.17	0.17 µm	0.8 µm	0.1 µm	0.4 µm
80	80.23	0.29%	%1	0.1 µm	0.4 µm
80	80.23	0.29%	%1	0.1 µm	0.4 µm
80	80.23	0.29%	%1	0.1 µm	0.4 µm
100	100.5	0.5%	%1	0.1 µm	0.4 µm
100	100.5	0.5%	%1	0.1 µm	0.4 µm
100	100.5	0.5%	%1	0.1 µm	0.4 µm

Acc. to table 3 in 2.1.3, diagonal measurement system is satisfactory.

3.4 Measurement on test cycle

Test cycle measurements can be seen from below table. According to Table 9, test cycle is satisfactory. All measurements are taken by same chronometer.

Table 9. Measurements for verification of test cycle.

Test Type	Passed time until max. test force (seconds)	Passed time in force application from initial force (seconds)	Extended Uncertainty of Chronometer (seconds)
HV0.1	5	12	±5
HV1	5	12	
HV5	6	12	
HV10	6	13	

3.5 Measurement on Reference Hardness Blocks (Indirect Calibration)

In order to verify hardness equipment, there shall be taken enough hardness measurements according to 2.2. All steps in 2.2 are followed and according to 2.2.1 and 2.2.2 repeatability and bias values are obtained.

Results and summary are shown in Table 10.

Table 10. Summary table of indirect verification.

Test Typ.	Reference	Taken Value	Avg. Value	Bias (%)	Max Permissible Bias (%)	Repeatability (%)	Max. Permissible Repeatability (%)	Std. deviation (HV)	Certificate Uncertainty (%)
HV0.1	750	745 738 744 737 742	741.2	-1.17	11	1.1	12	3.19	6.24
HV0.1	250.3	252 248 251 253 249	250.6	0.12	13.2	1.6	12	1.86	4.15
HV1	453.3	444 446 440 446 442	443.6	-2.14	4.5	1.35	8	2.33	1.87
HV1	736.3	724 722 718 721 722	721.4	-2.02	5.5	0.83	8	1.96	2.25
HV5	460.8	465 465 466 465 465	465.2	0.95	3	0.21	4	0.4	2
HV5	723.2	722 711 722 725 727	721.4	-0.25	3	2.22	4	5.34	2
HV10	436.9	444 445 445 445 444	444.6	1.76	3	0.22	4	0.49	2
HV10	736.2	717 722 721 721 722	720.6	-2.12	3	0.69	4	1.86	2.3

As it can be seen from Table 10, indirect verification of test machine is satisfactory. Additionally, Table 10 is also used for calculation of uncertainty calculations of this machine.

4. Uncertainty calculation

By indirect verification with reference blocks, the overall function of the hardness test machine is checked and the repeatability, as well as the deviation of the hardness test machine from the real hardness value (error), is determined.

The uncertainty of measurement of indirect verification of the hardness test machine is calculated according to below formula:

$$U_{HTM} = \sqrt{u_{CRM}^2 + {}_{CRM-D}^2 + {}_{H}^2 + {}_{ms}^2} \quad (5)$$

Where,

- u_{CRM} is the calibration uncertainty of hardness reference block acc. to its certificate for k=1.
- u_{CRM-D} hardness change because of drift. (It is negligible for this certified blocks).
- u_H is the standard uncertainty of the hardness testing machine when measuring the CRM.
- u_{ms} is the uncertainty due to the resolution of the diagonal measuring system of hardness testing machine. Both resolution of the length measurement indicating instrument and the optical resolution of the measuring microscope shall be considered.

Using results as shown in Table 10, for 453.3 HV1;

- U_{CRM} = 1.87 HV1 when k=2 ; 0.935 HV1 when k=1
- U_{CRM-D} is neglected because there was no previous measurement for this reference block
- $U_H = {(t * Sh)}/{\sqrt{n}}$ where constant t=1.14 from, n= 5 S_h (standard deviation) = 2.33 HV1, then U_H = -1.19
- $U_{ms} = {\delta ms}/{2\sqrt{3}}$ where $\delta ms = \sqrt{\delta or^2 + \delta ır^2}$ and $\delta or = {\lambda}/{2xNA}$

A is wavelength of yellow light (0.58µm) and NA is numerical aperture of the objective where 0.70 for x50 magnification and 0.25 for x10 magnification [5].

δ_{OR} : Optical resolution of the microscope objective.

δ_{IR} : Resolution of the display indicator of the measuring system which is 0.0001 mm.

Thus;

$\delta or = {0.00058\ mm}/{2x0.7\ mm}$ = 0.000414

$\delta ms = \sqrt{0.000414^2 + 0.0001^2}$ = 0.000426 mm

Then;

U_{ms} = 0.000426 mm / 2√3 = 0.000123 mm

4.1 Budget of uncertainty

Budget of uncertainty is shown in Table 11.

Table 11. Budget of uncertainty for 453.3 HV1.

Quantity X_p	Estimated value X_p	Standard uncertainty of measurement $U(X_p)$	Distribution type	Sensitivity coefficient C_p	Uncertainty contribution U_p (H) Hv1
u_{CRM}	453.3 HV1	0.935 HV1	NORMAL	1	0.935
u_H		1.19 HV1	NORMAL	1	1.19
u_{ms}		0.000123mm	Rectangular	-14165.6[a]	1.74
u_{CRM-D}	-	-	Triangular	1	0
Combined uncertainty of measurement, U_{HTM}					3.87
Expanded uncertainty of measurement, U_{HTM} (k=2)					7.74

a: $c=\frac{\partial H}{\partial d}$=-2 (H/d) and using the formula $HV = 0.189 * \frac{F}{d^2}$
Force is 9.807 N for HV1 and HV=453.3 d= 0.064
Then c= -2 (453.3/ 0.064) = -14165.6

The maximum deviation of the test machine can be calculated with below formula:

$$\Delta H_{HTMmax} = U_{HTM} + |b| = 7.74 \text{ HV1} + 2.14 \text{ HV1} = 9.88 \text{ HV1}$$

Where b is bias and comes from Table 10.

Additionally, same calculations can be applied to other reference blocks. For example, in this study there are also other measurements for 736.3 HV1 reference hardness block. By use of Eq. (5) and using results are shown in Table 10, for 736.3 HV1.

- U_{CRM} = 2.25 HV1 when k=2 ; 1.13 HV1 when k=1
- U_{CRM-D} is neglected because there was no previous measurement for this reference block
- $U_H = (t * Sh)/\sqrt{n}$ where t=1.14 , n= 5, S_h (std. dev.) = 1.96 HV1, then U_H= 1.50

Because the same objective is used when taking the measurement;

- $U_{ms} = 0.000426 \text{ mm} / 2\sqrt{3} = 0.000123 \text{ mm}$

If another objective would be used, then NA value also would be different.
Therefore, budget of uncertainty for 736.3 HV1 is shown in table 12.

Table 12 Budget of uncertainty for 736.3 HV1

Quantity X_p	Estimated value X_p	Standard uncertainty of measurement $U (X_p)$	Distribution type	Sensitivity coefficient C_p	Uncertainty contribution U_p (H) Hv1
u_{CRM}	736.3 HV1	1.13 HV1	NORMAL	1	1.13
u_H		1.50 HV1	NORMAL	1	1.50
u_{ms}		0.000123mm	Rectangular	-29334.66[a]	3.61
u_{CRM-D}	-	-	Triangular	1	0
Combined uncertainty of measurement, U_{HTM}					6.24
Expanded uncertainty of measurement, U_{HTM} (k=2)					12.48

a: c=$\frac{\partial H}{\partial d}$=-2 (H/d) and using the formula $HV = 0.189 * \frac{F}{d^2}$
Force is 9.807 N for HV1 and HV=736.3) d= 0.0502
Then c= -2 (736.3/ 0.0502) = -29334.66

5. Conclusion

In conclusion, it is necessary that effects of measurable and unmeasurable parameters which affect to performance of test equipment shall be checked when hardness test equipment is calibrated. Force, test mark monitoring system, indenter physical condition and geometry, test cycle directly affect test application. Uncertainty calculations can be made by use of only these parameters. However, in this situation some unpredictable problems such as geometrical damage on seat of test area might be kept out of coverage. In order to eliminate these kinds of risks and reveal the effects of unmeasured and unpredictable parameters, uncertainty calculations shall be made by use of reference hardness block [4].

Certified reference blocks shall not be only used for verification but also shall be used in order to make uncertainty calculation. This has a critical contribution in uncertainty budget. Another important point that shall be taken in consideration is objective choose. Used objective in determining mean diagonal of test marks is so important that affect uncertainty value. The reason of this is related to numerical aperture of objective. As it can be understood from table 11 and table 12, resolution of diagonal measurement system has a critical role in uncertainty budget. More increasing hardness values create more smaller marks on the image. This causes a relatively high uncertainty value in low magnifications. To obtain more precise results, higher magnifications with higher numerical apertures shall be preferred. Consequently, uncertainty values that are calculated by taking all of these into consideration shall be tabled and correlated with related test scales and magnifications.

6. References

1. https://www.industrialheating.com/ext/resources/Issues/2016/August/ih0816-mct-fig2-900.jpg
2. DIN EN ISO 6507-2, Metallic Materials, Vickers Hardness Test Part2: Verification and Calibration of Testing Machines
3. M.Bulut, *Sertlik Ölçme Cihazları Kalibrasyonu, 5*
4. M.Kuzu, VII. Ulusal Ölçüm Bilim Kongresi, *Metal sertliği test cihazlarının kalibrasyonu ve belirsizlik hesapları* , 517 (2008)
5. https://www.zeiss.com/microscopy/int/solutions/reference/basic-microscopy/numerical-aperture-and-resolution.html