APMP

TCM

Final Report of the Key Comparison of Vickers Hardness

APMP.M.H-K1b and APMP.M.H-K1c

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1. Introduction

CCM key comparison of Vickers hardness (CCM.H-K1) was held during 2001 to 2003 and the metrological equivalence of national standards among participated national metrology institutes (NMIs) was already published [1]. At that time, several NMIs took part in CCM Key comparison from APMP region but the equivalence of other NMIs in APMP were entrusted to the regional metrology organization (RMO), *i. e.*, APMP.

During the meeting of the APMP Working Group on Hardness on November, 2001 in Hanoi it was agreed to carry out a key comparison of Vickers Hardness Scales in which should participate the hardness laboratories of NMIs, and National Metrology Institute of Japan / National Institute of Advanced Industrial Science and Technology (NMIJ / AIST) was nominated as the pilot laboratory of the comparison.

This comparison APMP.H-K1 for Vickers hardness was started in 2003 and the measurement finished in 2005. The report is to demonstrate the equivalence of national standard for Vickers hardness worldwide. This comparison, APMP.H-K1 links to CIPM CCM Key comparison for Vickers hardness, CCM.H-K1 through the linking institute results.

2. Organization

The technical protocol was prepared by the pilot laboratory (Satoshi Takagi, NMIJ / AIST, Japan) following the CIPM guideline[2] and the technical protocol of the prior key comparison of CCM. The draft document of the technical protocol was circulated to the participating institutes and agreed before the comparison. The measurement was started in January, 2004 and finished in April 2005. The discussion on the results was mainly carried out via e-mail.

2.1 Participants

The list of participants is given in Table 2.1.

Table 2.1 List of participants

Country / economy	Laboratory	Contact
China	National Institute of Metrology (NIM) [a]	Dr. Ping Yang (Mr. He Li[b]) Bei San Huan Dong Lu 18, Beijing 100013 P. R. China
Chinese Taipei	Center for Measurement Standards / Industrial Technology Research Institute (CMS / ITRI)	Dr. Chung-Lin Wu (Dr. Fu-Lung Pan[b]) Bldg. 8, 321, Sec.2, Kuang Fu Rd, Hsinchu, Taiwan 30011
Hong Kong, China	The Government of The Hong Kong Special Administrative Region, Standards and Calibration Laboratory (SCL)	Mr. Raymond Leung (Mr. T. K. Chan[b]) 36/F, Immigration Tower, 7 Gloucester Road, Wanchai, Hong Kong
Japan (Pilot)	National Metrology Institute of Japan, National Institute of Advanced Science and Technology (NMIJ / AIST) [a]	Dr. Koichiro Hattori, (Dr. Satoshi Takagi[b]) [Coordinator] Tsukuba Central 3, 1-1-1 Umezono Tsukuba Ibaraki 305-8563, Japan
Korea	Korea Research Institute of Standards and Science (KRISS) [a]	Mr Nae-Hyung Tak (Dr. Gun-Woong Bahng[b]) P. O. Box 102, Yusong, Taejon 305-600, Korea

Thailand	National Institute of Metrology (Thailand) (NIMT)	Mr. Tassanai Sanponpute (Mr. Veera Tulasombut[b]) 3/4-5 Moo 3, Klong 5, Klong Luang, Pathumthani 12120 Thailand
Vietnam	Vietnam Metrology Institute (VMI)	Mr. Pham Thanh Ha (Mr. Vo Sanh[b]) 8- Hoang Quoc Viet Street, Cau giay Distr. Hanoi, Vietnam

[a] Linking laboratories to CCM Vickers key comparison (CCM.H-K1)

[b] Contact person in the beginning of this project

2.2 Timeline

The dates scheduled and actually measured by participants were listed in Table 2.2.

Table 2.2 Dates scheduled and actually measured by participants

Laboratory	Scheduled	Measured
NMIJ / AIST	Dec., 2003	Jan. , 2004
KRISS	Feb., 2004	Feb.,-Mar., 2004
NIM	May, 2004	May, 2004
SCL	Jun., 2004	Jul., 2004
CMS / ITRI	Jul, 2004	Aug., 2004
VMI	Aug., 2004	Oct.-Jan., 2004-2005
NIMT	Sep., 2004	Feb.- Apr., 2005
NMIJ / AIST	Oct., 2004	Apr., 2005

3. Transfer standards

One of the transfer standards used for this comparison is show in Fig. 3.1.

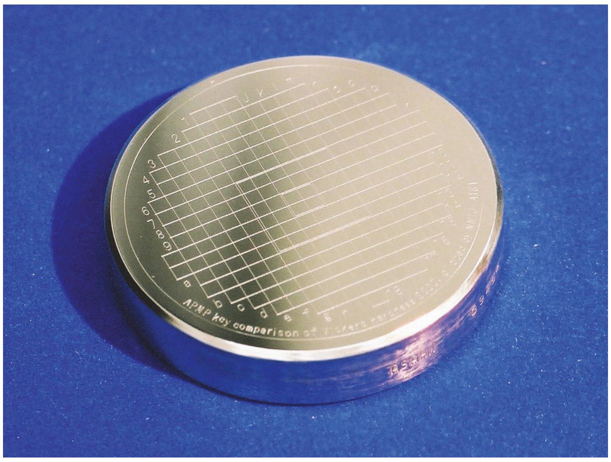

Fig. 3.1 Transfer standard used for the comparison.

In the key comparison, 3 hardness reference blocks at the hardness levels of 200 HV, 600 HV and 900 HV were used. The dimensions of the blocks are 65 mm in diameter and 15 mm in thickness. The blocks are manufactured as commercial products by Asahi Giken Co. Ltd., Japan. The measurement surface on the reference blocks was engraved a grid with 227 square segments (see Fig. 3.2). Each segment was used for single indentation for this comparison. The location of each segment is designated with the combination of a letter (a to r) and a number (1 to 18), which are engraved outside of the segments. The numbers define the position in *y*-direction (rows) and the letters define the position in *x*-direction (columns). These segments are divided into three sections bordered by double lines. 191 segments in the outside section (section A) were used for the measurements and 4 segments in the central section (section C) were used for the reference indentations. 32 segments in the intermediate section (section B) were reserved as the alternative places for wrong measurements.

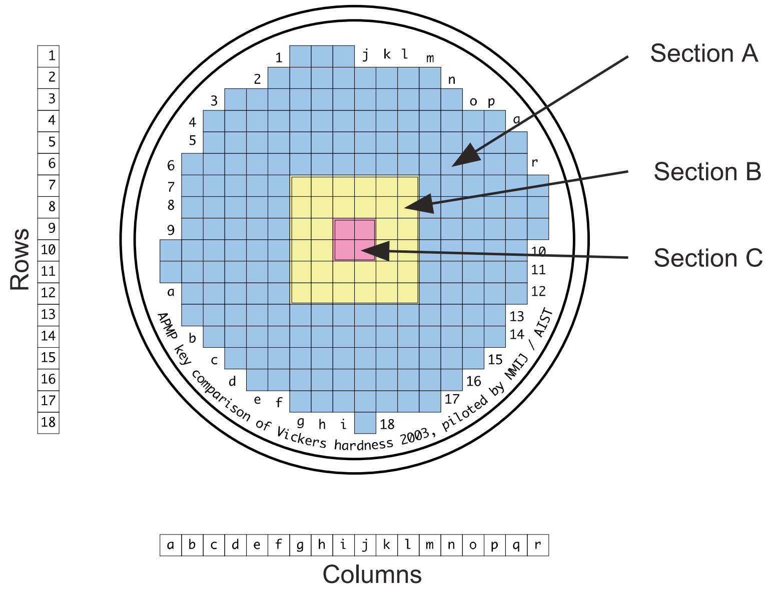

Fig. 3.2 Segment pattern on the measurement surface of reference blocks

In the outside section (section A), segments were divided equal-numbered 8 segment groups as shown in Fig. 3.3. 3 segments in each group were randomly assigned to each laboratory for three testing force, *i. e.*, HV 1, HV 10 and HV 30. As a result, each laboratory could measure hardness in randomly and equally distributed positions, so that the hardness distribution over the measurement surface would never affect to the average hardness evaluated by each laboratory.

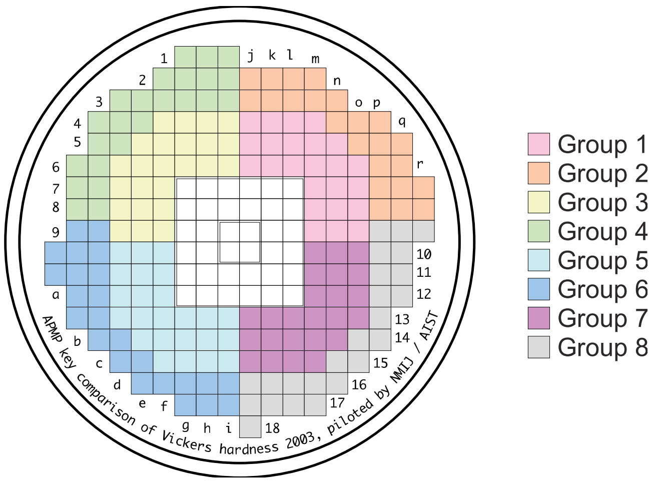

Fig. 3.3 Segment groups

In section C, 3 reference indentations were made. The locations of them are as follows.

(i10) for 9.807 N of testing force (HV 1)
(j9) for 98.07 N of testing force (HV 10)
(i9) for 294.2 N of testing force (HV 30)

Microscopic images of reference indentations are shown in Figs. 3.4 to 3.12. From the pictures, the bottoms of indentations don’t look clear. The reason is that the coordinator recommended to apply rust-prevent oil after the measurement and the oil seems to be still remained after the measurement surface was cleaned with solvent. The indentations are barrel shaped in 200 HV whereas they are pincushion shape in 900 HV. The shapes of 600 HV indentations are nearly square. In all pictures, the edges of indentations can be observed clearly and no damages to affect to the measurement are found.

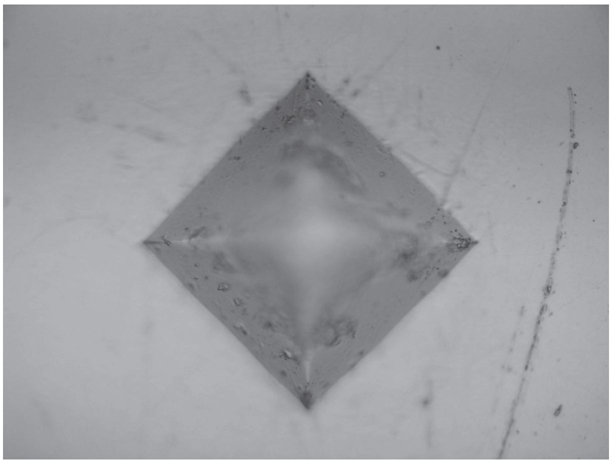

Fig. 3.4 Reference indentation of 200 HV 1 observed with 100× objective lens (NA=0.80).

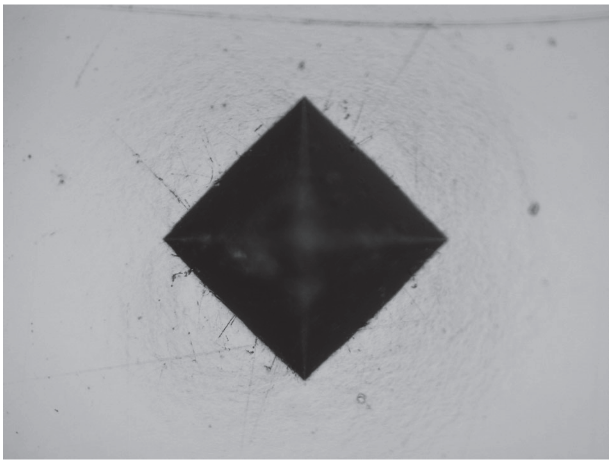

Fig. 3.5 Reference indentation of 200 HV 10 observed with 20× objective lens (NA=0.40).

Fig. 3.6 Reference indentation of 200 HV 30 observed with 20× objective lens (NA=0.40).

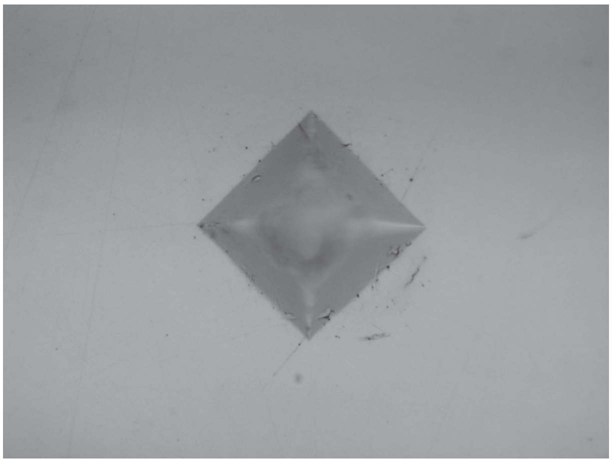

Fig. 3.7 Reference indentation of 600 HV 1 observed with 100× objective lens (NA=0.80).

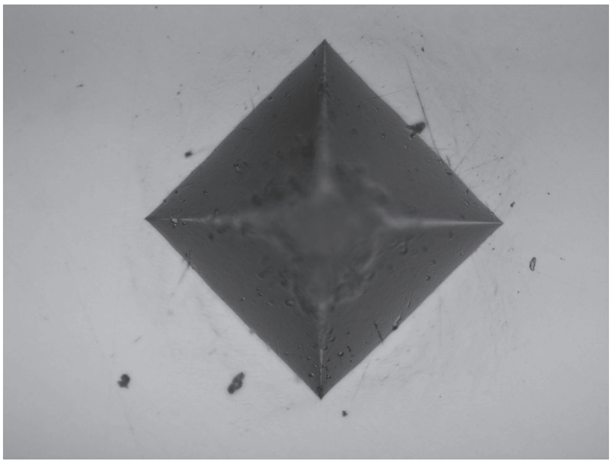

Fig. 3.8 Reference indentation of 600 HV 10 observed with 50× objective lens (NA=0.50).

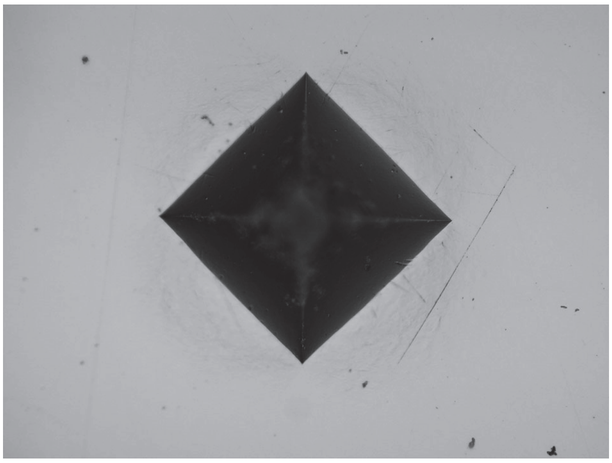

Fig. 3.9 Reference indentation of 600 HV 30 observed with 20× objective lens (NA=0.40).

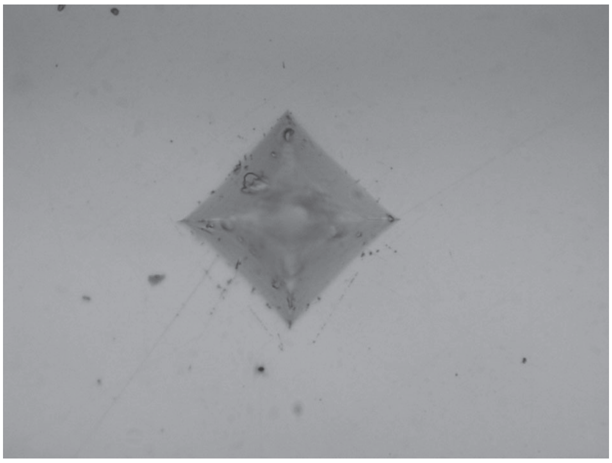

Fig. 3.10 Reference indentation of 900 HV 1 observed with 100× objective lens (NA=0.80).

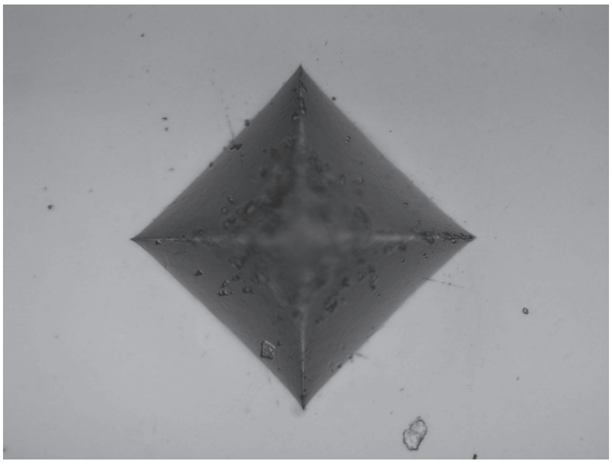

Fig. 3.11 Reference indentation of 900 HV 10 observed with 50× objective lens (NA=0.50).

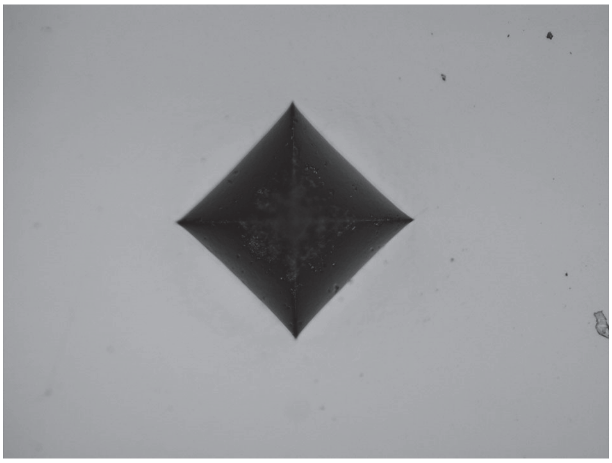

Fig. 3.12 Reference indentation of 900 HV 30 observed with 20× objective lens (NA=0.40).

The pilot laboratory made measurements of hardness and the dimension of reference indentations in the beginning and in the end of the key comparison in order to evaluate the stability of the used hardness reference blocks. The results will be described in section 6.2.

4. Measurement items

In this comparison, the participants measured hardness of 3 reference blocks in different hardness levels, *i. e.*, 200 HV, 600 HV and 900 HV with 3 different testing force, *i. e.*, 9.807 N, 98.07 N and 294.2 N according to the procedure specified in ISO 6507-1[3] and -3[4]. The average value of each reference block with each testing force was reported with estimated uncertainty.

In addition, the participants measured diagonal length of reference indentations made on each reference block with testing force of 9.807 N, 98.07 N and 294.2 N.

Because the measurement capability of some participants are limited, some laboratory reported only the results which they can able to measure. The detail of participated conditions of each laboratory is listed in Table 4.1.

Table 4.1 Overview of the measurements carried out by the participants

Institute	HV 1	HV 10	HV 30
NIM	×	×	×
CMS / ITRI	×	×	×
SCL	—	×	×
NMIJ / AIST	×	×	×
KRISS	×	×	×
NIMT	×	×	×
VMI	—	×	×

5. Methods of measurement

Before the measurements, each participating institute carried out the verification of their standard hardness testing machine according to ISO 6507-2[5] and -3. Items that participants were requested to report are

- the uncertainty of test force u_F,
- the uncertainty of length measuring device u_l,
- the angle between opposite faces of the indenter α (if possible),
- the length of the line of junction at the point of indenter c (if possible).

Each participant made on each hardness reference block 8 indentations in 8 groups of segments described in section 3. Since the levels of testing forces were 9.807 N (HV 1), 98.07 N (HV 10) and 294.2 N (HV 30), total number of indentations on each block was 24 for each participating institute if the institute was capable to measure all hardness scales. Each participating institute measured the diagonal lengths of the indentations according to the procedure described in ISO 6507-1. The number of 8 indentations allows to evaluate the stochastic deviations occurring during the measurements including the evaluation of the inhomogeneity of the hardness distribution on the hardness reference blocks. The same number of measurement is also employed in the CCM Vickers hardness key comparison, CCM.H.K-1. The diagonal lengths of the indentations in lateral and longitudinal direction ware measured 3 times. For each indentation, each participant reports

- the average value of the diagonal length in lateral direction,
- the average value of the diagonal length in longitudinal direction,
- the Vickers hardness calculated from the average of the altogether 6 diagonal lengths in lateral and longitudinal direction.

Any differences of the test procedure from ISO 6507-3 were requested to report. The participants used their own indenter for the measurements.

In addition, each participant measured the diagonal lengths of 3 reference indentations on each hardness reference block, which correspond to 3 levels of testing force. In this way it may be possible to separate the influence of the measuring system for the diagonal length from the overall uncertainty of the Vickers measurement.

There were no places to make extra indentations on reference blocks. However some participants might need additional indentations to stabilize their machine or to find suitable testing cycles. For such purposes one more block was prepared at each hardness level (practice block). There were no grids on these blocks and participants could use them as they like.

The locations of 24 indentations on each block for each participating institute were given in coordinates (the combination of a letter and a number). Each participating institute was informed of only the locations where they would make measurements so that the anonymity of each participating institute was guaranteed.

If there were some wrong measurements, the participants could register them in the data report in the fields for wrong measurements (designation „error“) and use the nearest free segment in the section B for alternative measurements.

Vickers hardness machines which were used by the participating laboratories are described shortly in Annex A.

6. Characteristics of the transfer standards

6.1 Uniformity of the reference blocks

Figs. 6.1 to 6.9 show the uniformity of hardness over the test surfaces of the reference blocks evaluated by all participants. In each chart, measured hardness values are plotted against the groups of test surface region described in Section 3. Therefore these charts represent hardness distribution over the test surface. As seen in these charts, no significant trends are however found. It suggested that the test surfaces of these reference blocks are uniform in hardness and there are no hardness distributions too. The uniformity of the reference blocks is listed in Table 6.1. It should be considered that the uniformity of the reference block and the stability of hardness testing machine cannot be separated in principle and the values in the list should be regarded as the combination of those two factors.

Table 6.1 Uniformity of the reference blocks evaluated by each laboratory

Hardness level, HV	Testing force, N	Average hardness range, HV	Standard deviation range, HV	Range of variation coefficient, %
200	9.807	209.2 to 215.6	0.70 to 3.32	0.33 to 1.58
	98.07	210.5 to 213.6	0.32 to 1.20	0.15 to 0.56
	294.2	210.5 to 213.4	0.22 to 1.01	0.10 to 0.48
600	9.807	597.1 to 617.1	2.97 to 5.21	0.50 to 0.84
	98.07	600.0 to 614.6	0.69 to 3.62	0.11 to 0.60
	294.2	601.1 to 611.2	0.84 to 2.93	0.14 to 0.48
900	9.807	897.9 to 922.0	0.26 to 5.58	0.26 to 0.60
	98.07	896.1 to 942.1	2.84 to 7.16	0.31 to 0.66
	294.2	896.7 to 930.7	1.99 to 4.48	0.22 to 0.50

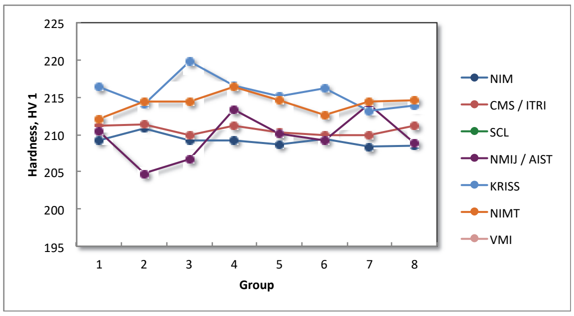

Fig. 6.1 Hardness distribution of 200 HV block evaluated by all participants in HV 1

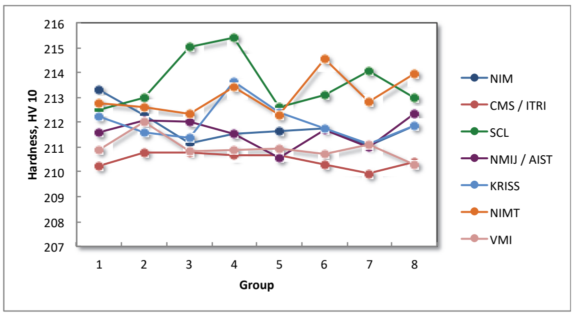

Fig. 6.2 Hardness distribution of 200 HV block evaluated by all participants in HV 10

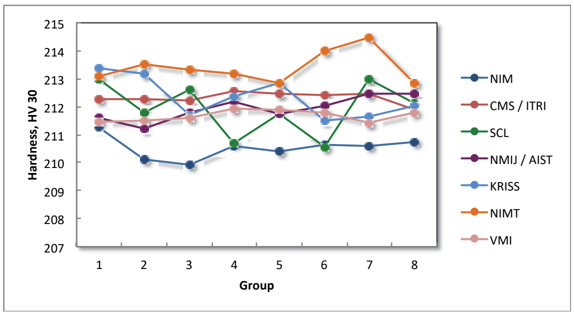

Fig. 6.3 Hardness distribution of 200 HV block evaluated by all participants in HV 30

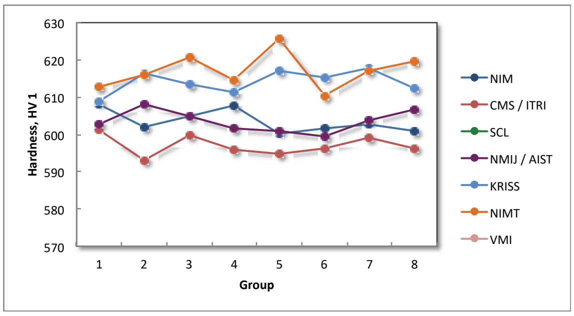

Fig. 6.4 Hardness distribution of 600 HV block evaluated by all participants in HV 1

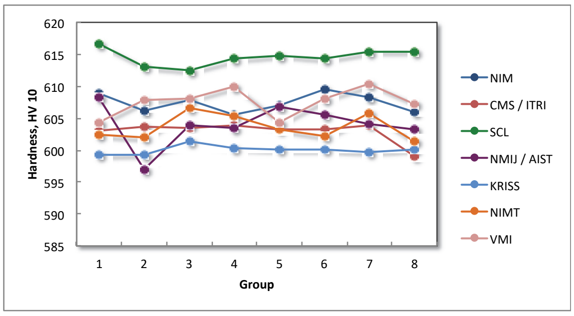

Fig. 6.5 Hardness distribution of 600 HV block evaluated by all participants in HV 10

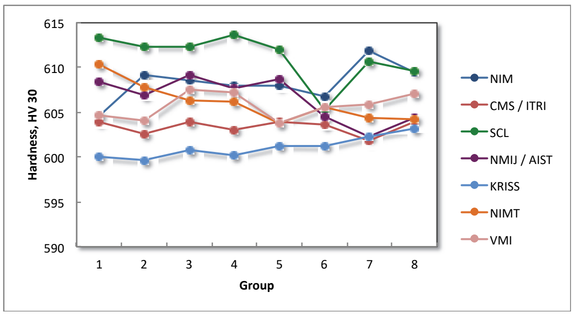

Fig. 6.6 Hardness distribution of 600 HV block evaluated by all participants in HV 30

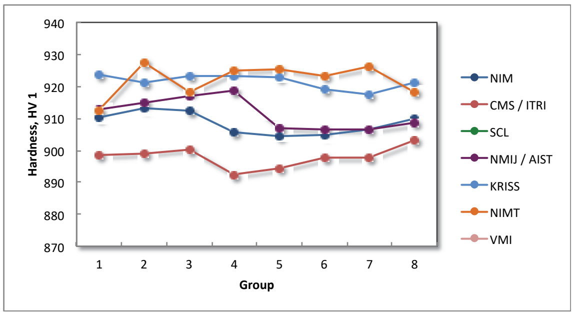

Fig. 6.7 Hardness distribution of 900 HV block evaluated by all participants in HV 1

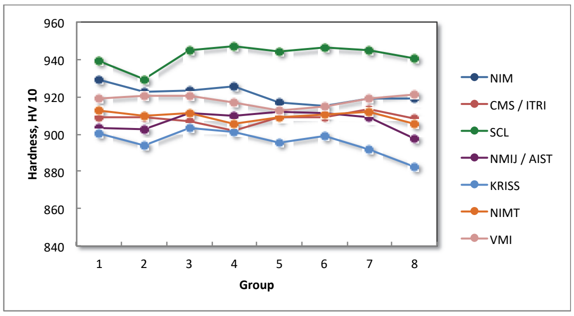

Fig. 6.8 Hardness distribution of 900 HV block evaluated by all participants in HV 10

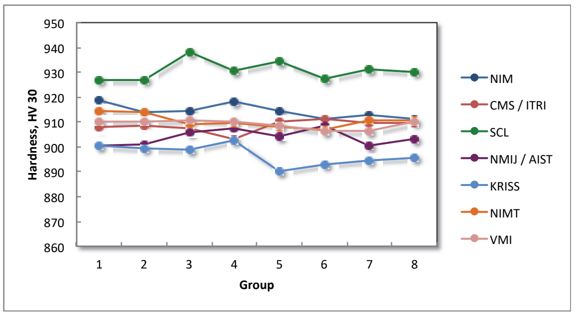

Fig. 6.9 Hardness distribution of 900 HV block evaluated by all participants in HV 30

Figs. 6.10 to 6.12 show the dependency of testing force on hardness of the reference blocks. As seen in these charts, no testing force dependency is found in 200 HV block. On the other hand, certain trends are observed in 600 HV and 900 HV blocks. Nevertheless, it never affects to the comparison results because every laboratory made measurements in the same condition.

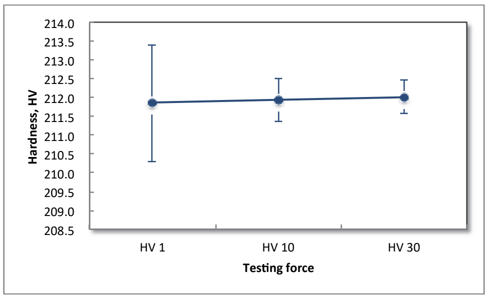

Fig. 6.10 Testing force dependency of hardness on 200HV block evaluated by all participants

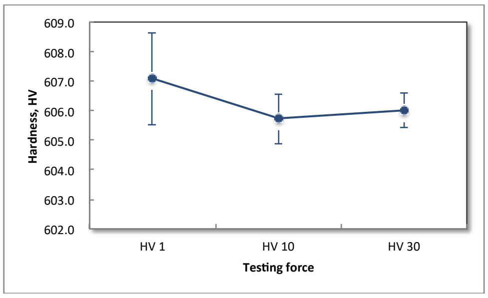

Fig. 6.11 Testing force dependency of hardness on 600HV block evaluated by all participants

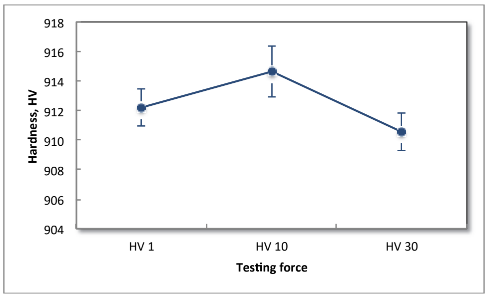

Fig. 6.12 Testing force dependency of hardness on 900HV block evaluated by all participants

6.2 Stability of the reference blocks

The stability of reference blocks was confirmed by the pilot laboratory in the beginning and in the end of the comparison. The results are shown in Table 6.2. The degrees of equivalence, i. e, the difference of hardness $d_1 = x_{ref} - x_1$ and its expanded uncertainty $U(d_1)$, were calculated assuming the measured values as reference values. As shown in this table, it can be concluded that those reference blocks are stable enough when the differences of measured values d_1 are compared with those uncertainties $U(d_1)$. Figs. 6.13 to 6.15 are the graphical representations of the stability of reference blocks. The error bars in those charts represent expanded uncertainties of measured average hardness. Those charts also indicate the reference blocks use for this comparison were stable.

Table 6.2 Measurement results of the hardness in the beginning and in the end of the comparison by the pilot laboratory

Hardness level, HV	Testing force, N	Measurements in the beginning, HV		Measurements in the end, HV		Degree of equivalence, HV	
		Average x_{ref}	Expanded uncertainty $U(x_{ref})$	Average x_1	Expanded uncertainty $U(x_1)$	Difference $d_1 = x_1 - x_{ref}$	Expanded Uncertainty $U(d_1)$
200	9.807	213.9	6.7	209.7	6.6	-4.2	9.4
	98.07	213.1	4.3	211.6	4.2	-1.5	6.0
	294.2	211.8	4.2	211.9	4.2	0.1	6.0
600	9.807	603.6	27.8	603.5	27.8	-0.1	39.3
	98.07	609.7	13.1	604.0	12.9	-5.6	18.4
	294.2	607.6	12.1	606.5	12.1	-1.1	17.2
900	9.807	913.8	49.7	911.4	49.5	-2.4	70.2
	98.07	908.2	21.8	907.0	21.8	-1.2	30.8
	294.2	904.9	18.1	903.9	18.1	-0.9	25.6

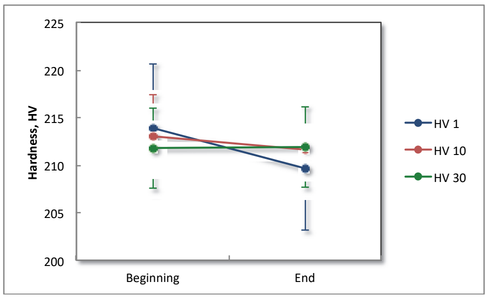

Fig. 6.13 Stability of reference blocks of 200 HV.

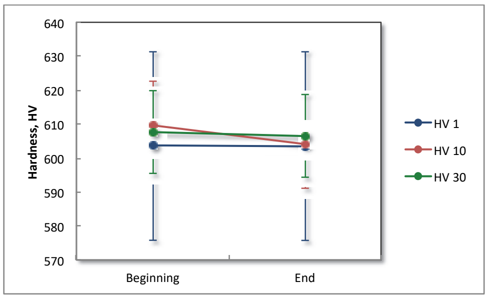

Fig. 6.14 Stability of reference blocks of 600 HV.

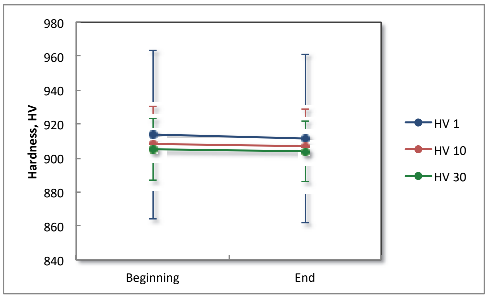

Fig. 6.15 Stability of reference blocks of 900 HV.

6.3 Stability of the reference indentations

The stability of reference indentations was also confirmed by the pilot laboratory in the beginning and in the end of the comparison. The results are shown in Table 6.3. Taking the same procedure with Table 6.2, the degrees of equivalence were calculated assuming the measured values as reference values. As shown in this table, it can be concluded that those reference indentations are also stable enough when the differences of measured values are compared with those uncertainties. Readers may think uncertainties written in the table are too large but it should be noted that the pilot laboratory estimated the uncertainty with not only the repeatability of measurement but the day-to-day variation of measurement by an operator (reproducibility). This is why the estimations are so conservative. The reader may also recognize that the measurement uncertainty for 900 HV 1 in the end of comparison is significantly improved between two measurements. The reason is that the pilot laboratory modified the standard hardness machine between two measurement and in the end of comparison the pilot laboratory used 100× objective lens instead of 40×. Figs. 6.16 to 6.18 are the graphical representations of the stability of reference indentations. The error bars in those charts represent expanded uncertainties of measured average diagonal length. Those charts also indicate the reference indentations used for this comparison were stable.

Table 6.3 Measurement results of the diagonal length of the reference indentations in the beginning and in the end of the comparison by the pilot laboratory

Hardness level, HV	Testing force, N	Measurements in the beginning, μm		Measurements in the end, μm		Degree of equivalence, μm	
		Average x_{ref}	Expanded uncertainty $U(x_{ref})$	Average x_1	Expanded uncertainty $U(x_1)$	Difference d_1	Expanded Uncertainty $U(d_1)$
200	9.807	93.798	1.272	93.973	1.268	0.175	1.542
	98.07	296.930	1.647	296.559	1.666	-0.371	1.989
	294.2	514.292	3.792	512.395	3.923	-1.897	4.529
600	9.807	56.233	1.272	56.281	1.266	0.049	1.544
	98.07	175.397	1.274	176.011	1.266	0.615	1.563
	294.2	303.269	1.653	303.759	1.671	0.490	1.979
900	9.807	45.471	1.265	45.653	0.557	0.182	1.289
	98.07	143.171	1.268	143.171	1.264	0.000	1.551
	294.2	248.009	1.672	247.740	1.671	-0.269	1.969

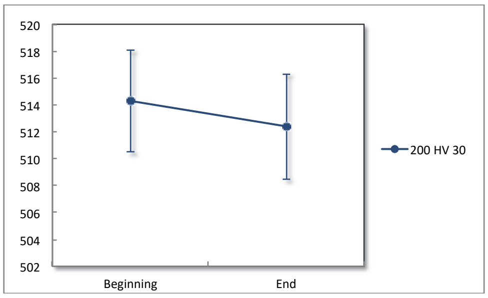

Fig. 6.16 Measurement results of the diagonal length of the reference indentations in the beginning and in the end of the comparison by the pilot laboratory with 10× objective.

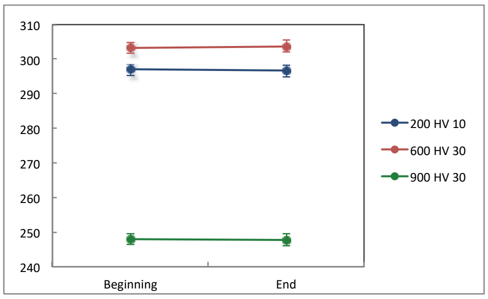

Fig. 6.17 Measurement results of the diagonal length of the reference indentations in the beginning and in the end of the comparison by the pilot laboratory with 20× objective.

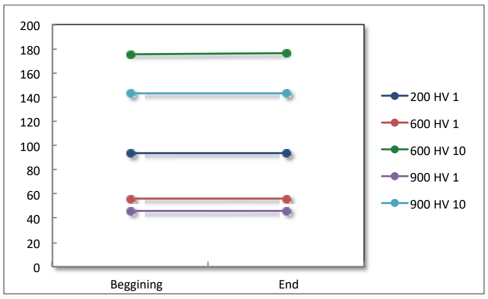

Fig. 6.18 Measurement results of the diagonal length of the reference indentations in the beginning and in the end of the comparison by the pilot laboratory with 40× or 100× objectives.

7. Measurement results

7.1 Measurement results of hardness of reference blocks

Reported results of hardness measurements and corresponding expanded uncertainties are listed in the tables 7.1 to 7.3.

Table 7.1 Measurement results of hardness at 200 HV level (unit in HV)

Institute	HV 1		HV 10		HV 30	
	Mean value	Expanded uncertainty	Mean value	Expanded uncertainty	Mean value	Expanded uncertainty
KRISS	215.7	1.2 %	212.0	0.8 %	212.3	0.5 %
NIM	209.0	1.3 %	211.8	0.6 %	210.6	0.5 %
SCL	—	—	213.6	2.8 %	212.0	2.8 %
CMS / ITRI	210.6	4.0 %	210.5	1.3 %	212.3	1.3 %
VMI	—	—	211.0	NR	211.7	NR
NIMT	212.1	3.2 %	212.8	2.0 %	213.1	2.0 %
NMIJ / AIST	209.7	3.2 %	211.6	2.0 %	211.9	2.0 %

TABLE 7.2 Measurement results of hardness at 600 HV level (unit in HV)

Institute	HV 1		HV 10		HV 30	
	Mean value	Expanded uncertainty	Mean value	Expanded uncertainty	Mean value	Expanded uncertainty
KRISS	614.2	2.0 %	600.0	1.3 %	601.1	0.8 %
NIM	603.7	2.2 %	606.6	1.1 %	608.3	0.9 %
SCL	—	—	614.6	2.6 %	611.2	2.6 %
CMS / ITRI	597.1	4.0 %	602.9	1.3 %	603.4	1.3 %
VMI	—	—	607.6	NR	605.7	NR
NIMT	617.1	4.7 %	603.6	2.1 %	606.1	2.0 %
NMIJ / AIST	603.5	4.7 %	604.0	2.1 %	606.5	2.0 %

Table 7.3 Measurement results of hardness at 900 HV level (unit in HV)

Institute	HV 1		HV 10		HV 30	
	Mean value	Expanded uncertainty	Mean value	Expanded uncertainty	Mean value	Expanded uncertainty
KRISS	921.8	2.4 %	896.1	1.6 %	896.9	1.0 %
NIM	908.1	2.7 %	921.1	1.3 %	914.2	1.1 %
SCL	—	—	942.1	3.2 %	930.7	3.2 %
CMS / ITRI	897.9	4.0 %	908.3	1.3 %	908.5	1.3 %
VMI	—	—	918.3	NR	909.1	NR
NIMT	912.6	5.5 %	912.7	2.4 %	914.6	2.0 %
NMIJ / AIST	911.4	5.5 %	907.0	2.4 %	903.9	2.0 %

NR: Not Reported.

7.2 Measurement results of the dimension of reference indentations

Reported results of dimension measurements of reference indentations are listed in the tables

7.4 to 7.6. Figures. 7.1 to 7.9 are the graphical representations of those results. Those charts show that there is some trend of reading diagonal length, *i. e.*, some laboratory always reads diagonal length larger but another laboratory always reads it smaller. This trend is quite significant if it is compared with the repeatability of readings.

Table 7.4 Measurement results of the diagonal length of reference indentations on the 200 HV reference block

Unit: μm

Institute	HV 1		HV 10		HV 30	
	Average	Repeatability	Average	Repeatability	Average	Repeatability
NIM	94.87	0.115	297.47	0.573	—	—
CMS / ITRI	93.30	0.172	297.60	1.358	512.38	1.082
SCL	—	—	295.08	0.649	510.25	0.304
NMIJ / AIST	93.97	0.099	296.56	0.244	512.40	0.432
KRISS	93.41	0.159	296.62	0.148	510.67	0.519
NIMT	93.31	0.184	296.27	0.665	510.37	0.404
VMI	—	—	297.17	0.115	512.18	0.084
Average	93.77	—	296.68	—	511.38	—

Table 7.5 Measurement results of the diagonal length of reference indentations on the 600 HV reference block

Unit: μm

Institute	HV 1		HV 10		HV 30	
	Average	Repeatability	Average	Repeatability	Average	Repeatability
NIM	55.58	0.115	175.95	0.061	305.08	0.109
CMS / ITRI	55.62	0.172	175.92	0.084	304.48	0.178
SCL	—	—	172.17	0.287	300.00	0.000
NMIJ / AIST	56.28	0.099	176.01	0.431	303.76	0.180
KRISS	55.83	0.159	175.35	0.230	303.38	0.381
NIMT	55.88	0.184	174.94	0.167	302.69	0.381
VMI	—	—	174.73	0.218	303.00	0.211
Average	55.84	—	175.01	—	303.20	—

Table 7.6 Measurement results of the diagonal length of reference indentations on the 900 HV reference block

Unit: μm

Institute	HV 1		HV 10		HV 30	
	Average	Repeatability	Average	Repeatability	Average	Repeatability
NIM	45.30	0.070	142.50	0.122	248.65	0.448
CMS / ITRI	45.44	0.118	143.05	0.208	247.43	0.057
SCL	—	—	140.17	0.287	243.83	0.287
NMIJ / AIST	45.65	0.154	143.17	0.228	247.74	0.181
KRISS	45.44	0.118	141.92	0.257	248.20	0.651
NIMT	45.43	0.255	141.88	0.435	246.83	0.252
VMI	—	—	142.20	0.378	247.55	0.061
Average	45.45	—	142.13	—	247.18	—

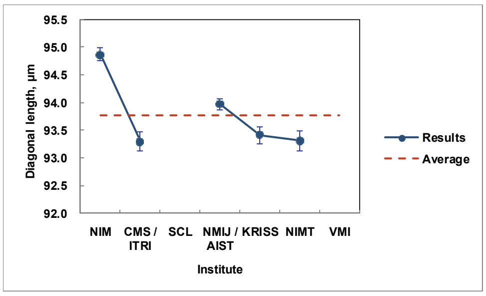

Fig. 7.1 Measurement results of the diagonal length of the reference indentation on the 200 HV 1 reference block.

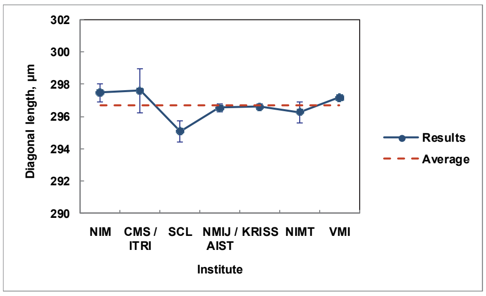

Fig. 7.2 Measurement results of the diagonal length of the reference indentation on the 200 HV 10 reference block.

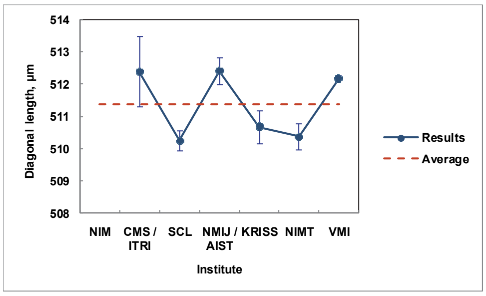

Fig. 7.3 Measurement results of the diagonal length of the reference indentation on the 200 HV 30 reference block.

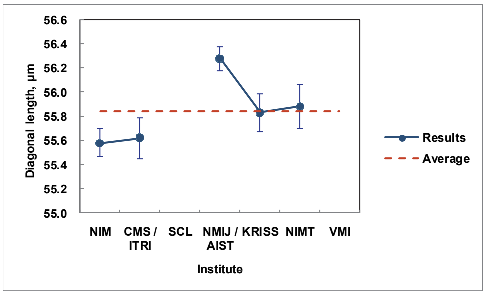

Fig. 7.4 Measurement results of the diagonal length of the reference indentation on the 600 HV 1 reference block.

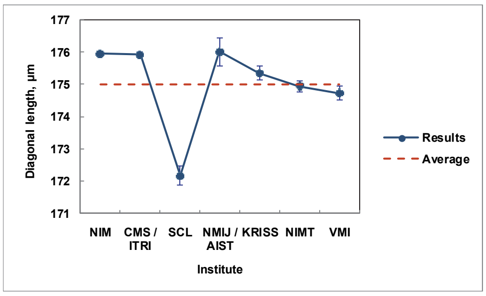

Fig. 7.5 Measurement results of the diagonal length of the reference indentation on the 600 HV 10 reference block.

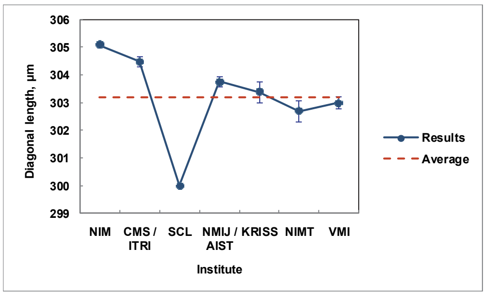

Fig. 7.6 Measurement results of the diagonal length of the reference indentation on the 600 HV 30 reference block.

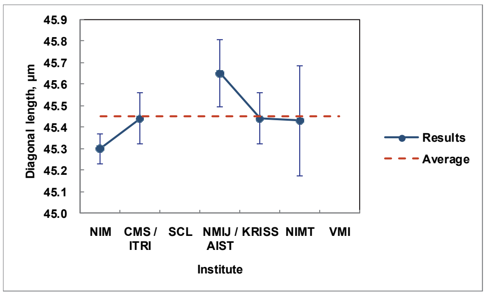

Fig. 7.7 Measurement results of the diagonal length of the reference indentation on the 900 HV 1 reference block.

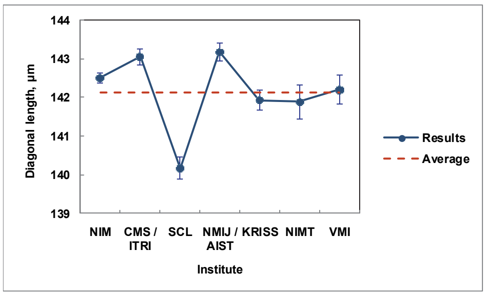

Fig. 7.8 Measurement results of the diagonal length of the reference indentation on the 900 HV 10 reference block.

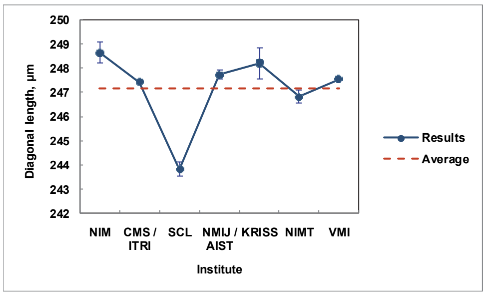

Fig. 7.9 Measurement results of the diagonal length of the reference indentation on the 900 HV 30 reference block.

8. Analysis

8.1 Linking to the Key comparison reference values (KCRVs) of CCM.H-K1

The APMP.H-K1 has been organized to link toward Key Comparison Reference Value (KCRV) of last CCM Key comparison (CCM KC) for Vickers hardness (CCM.H-K1). More strictly, HV 1 and HV 30 are linked to CCM.H-K1b and CCM.H-K1c, respectively. There are three institutes, KRISS, NIM and NMIJ, participated to the CCM KC. In the case of Vickers hardness, the largest uncertainty source is comes from the measurement of indentation diagonal length. Therefore, the reference values can be normalized with assuming the relation proportional to the hardness value. Comparison Reference Value (CRV) is calculated by the weighted mean of the results, x_i and it's uncertainties, $u(x_i)$ of three participated institutes to the CCM KC as:

$$x_{CRV} = \frac{x_1/u^2(x_1) + x_2/u^2(x_2) + x_3/u^2(x_3)}{1/u^2(x_1) + 1/u^2(x_2) + 1/u^2(x_3)} \quad (8.1)$$

The standard uncertainties of the CRVs $u(x_{ref})$ can be calculated by this equation,

$$u(x_{\mathrm{CRV}}) = \left[\frac{1}{u^2(x_1)} + \frac{1}{u^2(x_2)} + \frac{1}{u^2(x_3)}\right]^{-1/2} . \quad (8.2)$$

The results can be transferred using the ratio between KCRV and CRV as,

$$x_{\mathrm{CRV\_trans}} = \left(\frac{x_{\mathrm{CRV}}}{x_{\mathrm{KCRV}}}\right) x_{\mathrm{KCRV}} = r \cdot x_{\mathrm{KCRV}} \quad (8.3)$$

Where the *r* is the ratio of x_{KCRV} and x_{CRV}.
We use x_{CRV} as reference value that it directly links to the CCM Key Comparison Reference Value, x_{KCRV}, with expecting, x_{CRV}- $x_{\mathrm{CRV\_trans}}=0$. The uncertainty of reference value can be given by the combination of uncertainties of KCRV and of CRV. The reference value and its standard uncertainty is given by,

$$x_{\mathrm{ref}} = x_{\mathrm{CRV}} \equiv r \cdot x_{\mathrm{KCRV}}$$

$$u^2_{\mathrm{rel}}(x_{\mathrm{ref}}) = u^2_{\mathrm{rel}}(x_{\mathrm{CRV}}) + u^2_{\mathrm{rel}}(x_{\mathrm{KCRV}}) \quad (8.4)$$

Where, $u^2_{rel}(x_{CRV})$ and $u^2_{rel}(x_{KCRV})$ are the uncertainty of Comparison Reference value and Key Comparison Reference Value, respectively. The uncertainty with subscript "rel" indicates "relative" uncertainly. Reference values, expanded uncertainties with coverage factor k=2 and each uncertainty used to calculation of $u(x_{ref})$ are shown in Tables 8.1. Hardness scales, HV 1 and HV 30 were exercised in previous KC. Therefore, the complete linking toward CCM KC can be found only in these hardness scales. For HV 10 scale, the relative uncertainty, $u_{rel}(x_{ref})$ is calculated only by the $u_{rel}(x_{CRV})$.

TABLE 8.1 Reference Value and Expanded uncertainty.

Hardness scale	Reference value x_{ref}, HV	Relative expanded uncertainty $U(x_{ref})$, %	Relative uncertainty used.	
			U_{CRV}, %	U_{KCRV}, %[1]
200 HV 1	212.15	1.23	0.86	0.87
200 HV 10	211.86	0.47	0.47	−
200 HV 30	211.46	0.90	0.36	0.82
600 HV 1	608.93	1.80	1.39	1.14
600 HV 10	603.75	0.78	0.78	−
600 HV 30	604.40	1.12	0.57	0.96
900 HV 1	914.15	2.54	1.54	2.02
900 HV 10	912.25	0.85	0.85	−
900 HV 30	905.93	1.51	0.63	1.37

8.2 Degrees of equivalence

Degree of equivalence of the comparison is expressed with two parameters, *i. e.*, deviation from KCRV,

$$d_i = (x_i - x_{ref}) \tag{8.5}$$

its expanded uncertainty,

$$U(d_i) = k \cdot \left[u^2(x_i) + u^2(x_{ref})\right]^{\frac{1}{2}} \cdot \tag{8.6}$$

And the E_n number,

$$E_n = d_i / U(d_i) \tag{8.7}$$

Degree of Equivalence: unilateral analysis is shown in Table 8.2 and corresponding E_n number is shown in Table 8.3. The graphical represents of results are shown in figs. 8.1 to 8.9.

Table 8.2 Unilateral analysis of Degrees of equivalence, in %.

Institute	DoE	200 HV level			600 HV level			900 HV level		
		HV 1	HV 10*	HV 30	HV 1	HV 10*	HV 30	HV 1	HV 10*	HV 30
KRISS	d_i, %	1.68	0.07	0.39	0.87	-0.62	-0.55	0.84	-1.77	-1.00
	$U(d_i)$, %	1.74	0.93	1.03	2.65	1.49	1.37	3.46	1.84	1.80
NIM	d_i, %	-1.48	-0.03	-0.41	-0.86	0.47	0.65	-0.66	0.97	0.91
	$U(d_i)$, %	1.79	0.76	1.03	2.84	1.35	1.44	3.71	1.55	1.87
SCL	d_i, %	—	0.82	0.25	—	1.80	1.13	—	3.27	2.73
	$U(d_i)$, %	—	2.85	2.97	—	2.72	2.85	—	3.30	3.56
CMS / ITRI	d_i, %	-0.73	-0.64	0.39	-1.94	-0.14	-0.17	-1.78	-0.43	0.28
	$U(d_i)$, %	4.18	1.38	1.58	4.39	1.51	1.71	4.74	1.55	1.99
VMI	d_i, %	—	-0.41	0.11	—	0.64	0.22	—	0.66	0.35
	$U(d_i)$, %	—	NR	NR	—	NR	NR	—	NR	NR
NIMT	d_i, %	-0.02	0.44	0.77	1.34	-0.03	0.28	-0.17	0.05	0.96
	$U(d_i)$, %	3.38	2.06	2.19	4.99	2.28	2.29	6.02	2.55	2.50
NMIJ / AIST	d_i, %	-1.15	-0.12	0.21	-0.89	0.04	0.35	-0.30	-0.58	-0.22
	$U(d_i)$, %	3.38	2.06	2.19	4.99	2.28	2.29	6.02	2.55	2.50

NOTE1: Underlined institutes are whose results used to calculating the reference value.

NOTE2: NR (Not Reported the uncertainty).

NOTE3: *The result only in this comparison is indicated in HV 10, because HV 10 is not exercised in previous KC (CCM.H-K1).

Table 8.3 E_n number: unilateral analysis

Institute	200 HV level			600 HV level			900 HV level		
	HV 1	HV 10	HV 30	HV 1	HV 10	HV 30	HV 1	HV 10	HV 30
KRISS	0.96	0.07	0.38	0.33	-0.42	-0.40	0.24	-0.96	-0.55
NIM	-0.83	0.04	-0.40	-0.30	0.35	0.45	-0.18	0.62	0.49
SCL	—	0.29	0.09	—	0.66	0.40	—	0.99	0.77
CMS / ITRI	-0.17	-0.46	0.25	-0.44	-0.09	-0.10	-0.38	-0.28	0.14
VMI	—	NR	NR	—	NR	NR	—	NR	NR
NIMT	-0.01	0.22	0.35	0.27	-0.01	0.12	-0.03	0.02	0.38
NMIJ / AIST	-0.34	-0.06	0.09	-0.18	0.02	0.15	-0.05	-0.23	-0.09

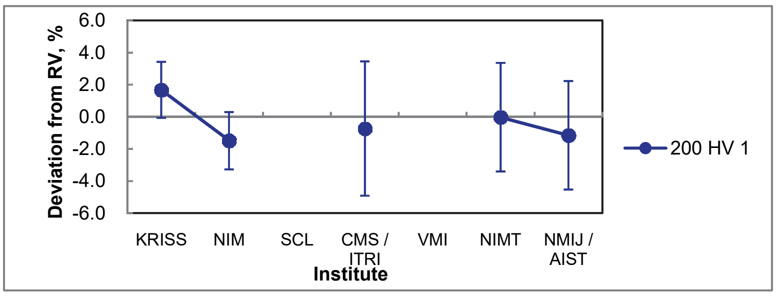

Fig. 8.1 Comparison Result of Hardness at 200 HV 1

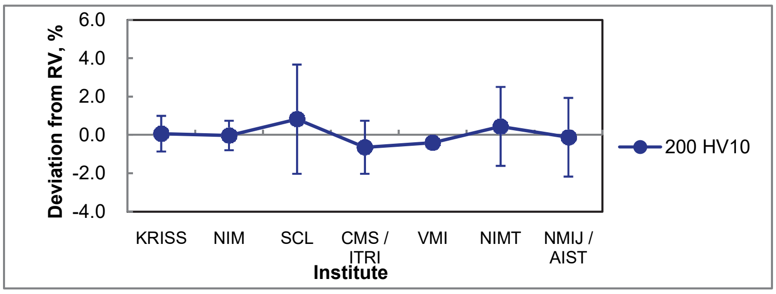

Fig. 8.2 Comparison Result of Hardness at 200 HV 10

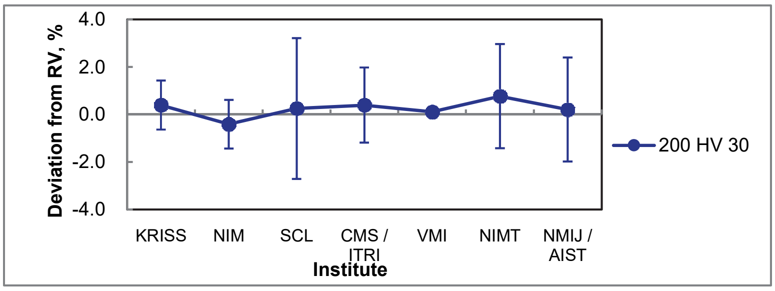

Fig. 8.3 Comparison Result of Hardness at 200 HV 30

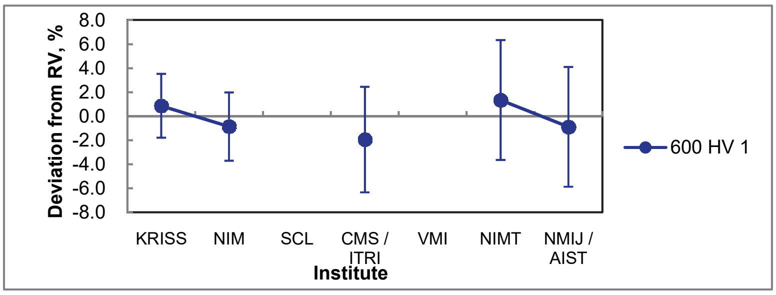

Fig. 8.4 Comparison Result of Hardness at 600 HV 1

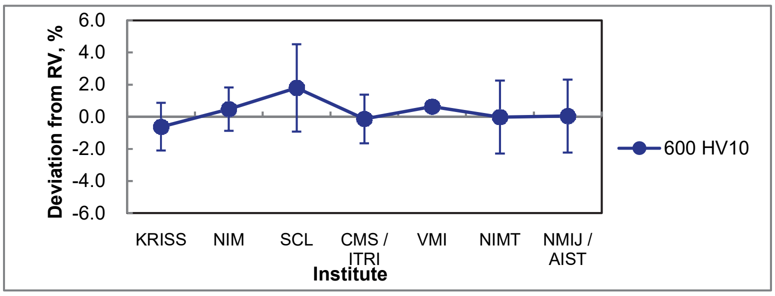

Fig. 8.5 Comparison Result of Hardness at 600 HV 10

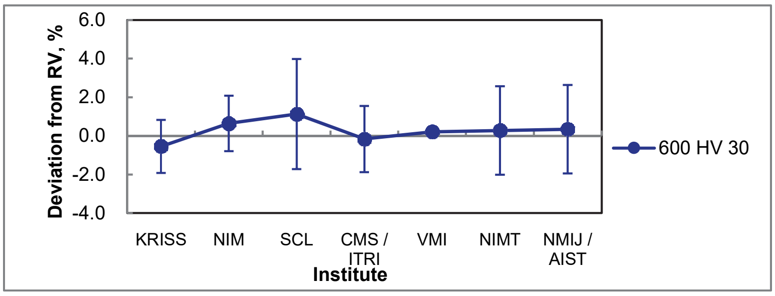

Fig. 8.6 Comparison Result of Hardness at 600 HV 30

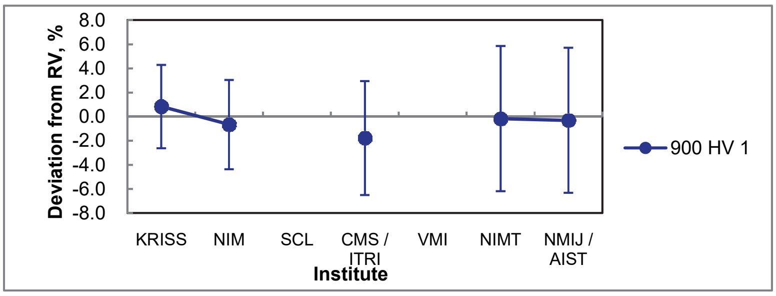

Fig. 8.7 Comparison Result of Hardness at 900 HV 1

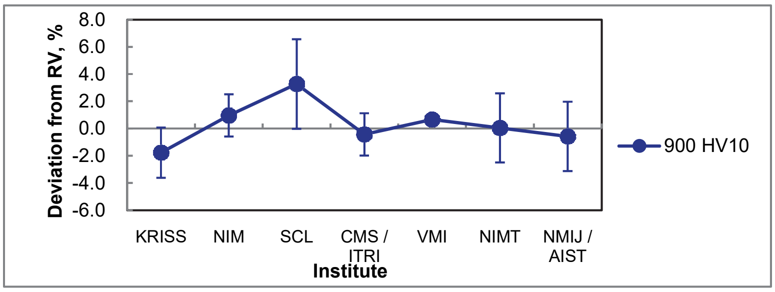

Fig. 8.8 Comparison Result of Hardness at 900 HV 10

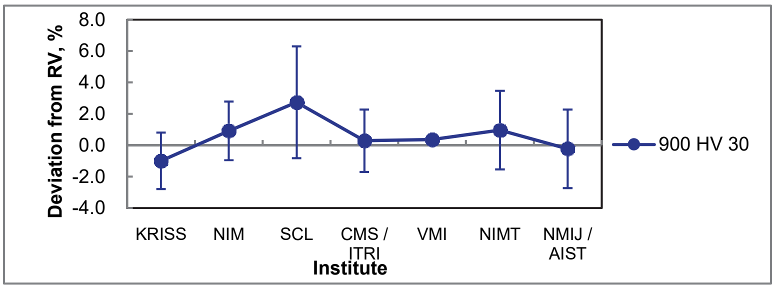

Fig. 8.9 Comparison Result of Hardness at 900 HV 30

8.3 Results summary: APMP.M.H-K1b (Vickers 1)

Table 8.4 Unilateral analysis of Degrees of equivalence, in HV, for Vickers hardness HV 1.

Institute	200 HV 1		600 HV 1		900 HV 1	
	d_i, HV	$U(d_i)$, HV	d_i, HV	$U(d_i)$, HV	d_i, HV	$U(d_i)$, HV
KRISS	3.6	3.7	5.3	16.2	7.7	31.6
NIM	-3.1	3.8	-5.2	17.3	-6.0	33.9
CMS / ITRI	-1.5	8.9	-11.8	26.7	-16.2	43.3
NIMT	0.0	7.2	8.2	30.4	-1.5	55.1
NMIJ / AIST	-2.4	7.2	-5.4	30.4	-2.7	55.1

NOTE: Underlined institutes are whose results used to calculating the reference value.

Table 8.5 Unilateral analysis of Degrees of equivalence, in %, for Vickers hardness HV 1.

Institute	200 HV 1		600 HV 1		900 HV 1	
	d_i, %	$U(d_i)$, %	d_i, %	$U(d_i)$, %	d_i, %	$U(d_i)$, %
KRISS	1.68	1.74	0.87	2.65	0.84	3.46
NIM	-1.48	1.79	-0.86	2.84	-0.66	3.71
CMS / ITRI	-0.73	4.18	-1.94	4.39	-1.78	4.74
NIMT	-0.02	3.38	1.34	4.99	-0.17	6.02
NMIJ / AIST	-1.15	3.38	-0.89	4.99	-0.30	6.02

NOTE: Underlined institutes are whose results used to calculating the reference value.

Table 8.6 E_n number: unilateral analysis, for Vickers hardness HV1.

Institute	200 HV 1	600 HV 1	900 HV 1
KRISS	0.96	0.33	0.24
NIM	-0.83	-0.30	-0.18
CMS / ITRI	-0.17	-0.44	-0.38
NIMT	-0.01	0.27	-0.03
NMIJ / AIST	-0.34	-0.18	-0.05

8.4 Results summary: APMP.M.H-K1c (Vickers 30)

Table 8.7 Unilateral analysis of Degrees of equivalence, in HV, for Vickers hardness HV 30.

Institute	200 HV 30		600 HV 30		900 HV 30	
	d_i, HV	$U(d_i)$, HV	d_i, HV	$U(d_i)$, HV	d_i, HV	$U(d_i)$, HV
KRISS	0.8	2.2	-3.3	8.3	-9.0	16.3
NIM	-0.9	2.2	3.9	8.7	8.3	16.9
SCL	0.5	6.3	6.8	17.2	24.8	32.2
CMS / ITRI	0.8	3.3	-1.0	10.4	2.6	18.0
NIMT	1.6	4.6	1.7	13.8	8.7	22.7
NMIJ / AIST	0.4	4.6	2.1	13.8	-2.0	22.7

NOTE: Underlined institutes are whose results used to calculating the reference value.

Table 8.8 Unilateral analysis of Degrees of equivalence, in %, for Vickers hardness HV 30.

Institute	200 HV 30		600 HV 30		900 HV 30	
	d_i, %	$U(d_i)$, %	d_i, %	$U(d_i)$, %	d_i, %	$U(d_i)$, %
KRISS	0.39	1.03	-0.55	1.37	-1.00	1.80
NIM	-0.41	1.03	0.65	1.44	0.91	1.87
SCL	0.25	2.97	1.13	2.85	2.73	3.56
CMS / ITRI	0.39	1.58	-0.17	1.71	0.28	1.99
NIMT	0.77	2.19	0.28	2.29	0.96	2.50
NMIJ / AIST	0.21	2.19	0.35	2.29	-0.22	2.50

NOTE: Underlined institutes are whose results used to calculating the reference value.

Table 8.9 E_n number: unilateral analysis, for Vickers hardness HV 30.

Institute	200 HV 30	600 HV 30	900 HV 30
KRISS	0.38	-0.40	-0.55
NIM	-0.40	0.45	0.49
SCL	0.09	0.40	0.77
CMS / ITRI	0.25	-0.10	0.14
NIMT	0.35	0.12	0.38
NMIJ / AIST	0.09	0.15	-0.09

9. Verification of standard hardness machines of participants

Verification results of each participating laboratory are described in Annex B.

The participants were also requested to report their uncertainty of measurement with their measurement results. The procedure of uncertainty of measurement depend on each participant and the results are also described in Annex B.

10. Conclusions

The comparison was successfully finished. The comparison item is Vickers hardness for 200 HV 1, 200 HV 10, 200 HV 30, 600 HV 1, 600 HV 10, 600 HV 30, 900 HV 1, 900 HV 10, and 900 HV 30.

Through this comparison, the results for 200 HV 1, 600 HV 1 and 900 HV 1 scales are linked to CCM.H-K1b, and the results for 200 HV 30, 600 HV 30 and 900 HV 30 scales are linked to CCM.H-K1c.

The reference values are calculated by using weighted mean from the results of KRISS, NIM and NMIJ, participated institutes to CCM.H-K1. The Degree of Equivalence (DoE) was indicated by the relative deviation from reference values. The measurement results show good agreements in this key comparison.

References

[1] K. Herrmann, “CCM Vickers key comparison Final Report”, *Metrologia*, **43**, Tech. Suppl., 07010 (2006).

[2] “Guidelines for CIPM key comparisons”, CIPM (1999).

[3] ISO 6507-1: 1997, “Metallic materials - Vickers hardness test – Part 1: Test method”.

[4] ISO 6507-3: 1997, “Metallic materials - Vickers hardness test – Part 3: Calibration of reference blocks”.

[5] ISO 6507-2: 1997, “Metallic materials - Vickers hardness test – Part 2: Verification of testing machines”.

[6] M. G. Cox, “The evaluation of key comparison data”, *Metrologia*, **39** (2002) 589 – 595.

Annex A. Description of testing machines of the participants

A.1 NIM (China)

Primary standard machine of Vickers hardness which is used for HV30

1. With deadweight and hydraulic loading system.
2. Test force duration: 15 sec.
3. Optical microscope
   Magnification: 200 times
4. Manufacture of the measuring device: China
   Manufacture of the indenter: Germany

Primary standard machine of Vickers hardness which is used for HV10

1. With deadweight and hydraulic loading system.
2. Test force duration: 15 sec.
3. Optical microscope
   Magnification: 400 time
4. Manufacture of the measuring device: China
   Manufacture of the indenter: Germany

Primary standard machine of Vickers micro-hardness which is used for HV1

1. With deadweight loading system.
2. Test force duration: 15 sec.
3. Optical microscope
   Magnification: 640 times
4. Manufacture of the measuring device: China
   Manufacture of the indenter: China

A.2 CMS / ITRI (Chinese Taipei)

General information of the Vickers hardness testing machine:

Manufacturer: Akashi Co., Japan
Model: SHT-41
Mfg-Y/M: 2001/06

Information of optical system (manufacturer, magnifications, numerical apertures):

Manufacturer: Nikon
Magnifications (numerical apertures): (a) 10×(0.25), (b) 20×(0.40), (c) 40×(0.65)

Information of the indenter (manufacturer):

Tokyo Diamond Tools Mfg. Co., Ltd.

Information of the testing cycle:

Symbol	The duration of application of the test time (s)	Time for application of the test force (s)
HV1	13	4
HV10	13	7 to 8
HV30	13	7 to 8

A.3 SCL (Hong Kong, China)

General information of the Vickers hardness testing machine:

Manufacturer: Fords Industrial Products Limited
Model no.: 2002
Serial no.: HTM 0579/90

Information of optical system (manufacturer, magnifications, numerical apertures):

Manufacturer: Vision Engineering Limited
Model no.: -
Dynascope serial no.: 9161
Magnification: -
Numerical aperture: -
Ocular resolution: 0.001 mm

Information of the indenter (manufacturer):

Manufacturer: Fords Industrial Products Limited
Model no.: -
Vickers diamond indenter serial no.: FIP509

Information of the testing cycle:

Hardness level	Application of test force	Time value (s)	Expanded uncertainty (s)	Coverage factor
HV10	Time from initial application of test force to reach full force	2.0	0.5	2.01
	Duration of test force	14.3	0.5	2.20
HV30	Time from initial application of test force to reach full force	2.6	0.5	2.01
	Duration of test force	14.1	0.5	2.05

A.4 NMIJ / AIST (Japan)

General information of the Vickers hardness testing machine:

Manufacturer: Akashi Corp. (Currently Mitutoyo Corp.)
Model no.: SHT-41 (modified by NMIJ)
Serial no.: 30033

Lever and deadweight loading system
Built-in measuring microscope with multiple objective lenses changeable with a revolver

Information of optical system (manufacturer, magnifications, numerical apertures):

Objectives:

Manufacturer, magnification and numerical aperture:

5×/0.10, Akashi
10×/0.25, Akashi
20×/0.40, Akashi
40×/0.65, Akashi
100×/0.90, Nikon

Ocular micrometer:

Manufacturer: Akashi Corp. (Currently Mitutoyo Corp.)
Magnification: 10×
Electrical readout system with a rotary encoder

Information of the indenter (manufacturer):

Manufacturer: Tokyo diamond manufacturing Co.
Serial no.: 43215

Information of the testing cycle:

Testing cycles of the measurements for this comparison are listed following table.

Table A.4.1 Testing cycles

Hardness level	Indentation speed, μm/s	Duration of testing force, s
200 HV 1	3	13
200 HV 10	7	
200 HV 30	17	
600 HV 1	4	
600 HV 10	10	
600 HV 30	25	
900 HV 1	3	
900 HV 10	9	
900 HV 30	19	

A.5 KRISS (Korea)

General information of the Vickers hardness testing machine:

For 9.807 N :

Machine type : Weight type
Model : Akashi MVK-1
Measuring Device: optical microscope with manual measuring system by rotary encoder

For 98.07 N to 294.2 N :

Machine type : Lever type
Model : Akashi SHT-41
Measuring Device : CCD camera(Auto reading system)

Information of optical system (manufacturer, magnifications, numerical apertures):

Manufacturer : Akashi, Japan
Magnification and numerical apertures :
For 9.806 N : ×40 (0.65)
For 98.07 N to 294.2 N : ×20 (0.40), ×10 (0.25)

Information of the indenter (manufacturer):

Osaka Diamond

Information of the testing cycle:

1) Test force application time : 4 s
2) Test force dwell time : 10 s

A.6 NIMT (Thailand)

General information of the Vickers hardness testing machine:

Akashi, Model: SHT-41, S/N: 50045, for test force > 1 kgf to 50 kgf
Bareiss, Model: V-test, S/N: 6020/03, for test force ≤ 1 kgf

Information of optical system (manufacturer, magnifications, numerical apertures):

Mitutoyo
Model: HM-124
Magnifications: 10× 0.25NA, 20× 0.4NA, 50× 0.55NA, 100× 0.8NA
Olympus
Model: BH2-UMA
Magnifications: 20× 0.46NA, 40× 0.65 NA, 50× 0.45NA, 100× 0.8 NA

Information of the indenter (manufacturer):

Tokyo Diamond, S/N TDS41777, for test force > 1 kgf
Bareiss, S/N 5079, for test force ≤ 1 kgf

Information of the testing cycle

Loading speed: 60 μm/s ± 1 μm/s for test force > 1 kgf
Time for application of the test force: < 1 s for test force ≤ 1 kgf
Duration of application of the test force: 14 s ± 0.5 s

A.7 VMI (Vietnam)

General information of the Vickers hardness testing machine:

Type of machine: HNG - 250, Ser No. 01/78
A deadweight Stadard machine for HR, HV and HB 187,5
Made in Germany 1978
Forces: 10 , 30 , 50, 60, 62,5; 100; 120; 125; 150; 187,5; 250 kgf

Information of optical system (manufacturer, magnifications, numerical apertures):

The optical device on small load HV tester was used.

1. Optical Ser. No. 84902, magnitude 200×, measuring range (0 – 0.6) mm, division 0,001 mm, on the type of the hardness tester KL 2

2. Optical Ser. No. 7306, magnitude 100× and 500×, measuring range (0 – 0.6) mm, division 0,001 mm, on the type of the hardness tester MINILOAD

Information of the indenter (manufacturer):

Indenter No. 4108
Made in Germany 1978

Information of the testing cycle:

The time from the initial application of the force until the full test force: 4 s
The duration of the test force: 13 s

Annex B. Verification results of standard hardness testing machines of the participants

B.1 NIM (China)

B.1.1 Results of the verification

Results of the verification of standard hardness testing machine is shown in Table B.1.1.

Table B.1.1 Verification results

Item	Test force	Length measuring device	Angle between opposite faces of the intender (if possible)	Length of the line of conjunction at the point of the indenter (if possible)
Symbol	u_F [N]	u_l [μm]	α [degree]	c [μm]
Value[a]	HV 30: ≤ 0.1% HV 10: ≤ 0.1% HV 1: ≤ 0.14%	HV30: 1.1 HV10: 0.7 HV1: 0.4	HV30: 0.02º(α=136º1´) HV10: 0.02º(α=136º1´) HV1: 0.1º(α=135º54´18˝)	HV30: 0.6 HV10: 0.6 HV1: 0.5

[a] Maximum permissible error

B.1.2 Uncertainty evaluation

According to the table 6 and 7 of the draft of EA: “Guideline for the estimation of the uncertainty of measurement of Brinell and Vickers hardness scales”, the result of uncertainty evaluation are as follows.

Table B.1.2 Sensitivity coefficients

Source	Sensitivity coefficient	Unit
Test force	$\frac{\partial H}{\partial F} = \frac{H}{F}$	HV/N
Measuring device	$\frac{\partial H}{\partial d} = -2\frac{H}{d}$	HV/μm
Angle between opposite faces of the indenter	$\frac{\partial H}{\partial \alpha} = \frac{H}{2\tan(\alpha/2)} \cdot \frac{\pi}{180}$	HV/º
Line of conjunction of the indenter	$\frac{\partial H}{\partial c} = -\frac{\sqrt{2}}{d} \cdot H$	HV/μm

Table B.1.3 Evaluation of the uncertainty for HV30

Influencing Quantity x_i	Value a_i	Variances $(\Delta HV_i/HV_i)^2$		
		900 HV 30 (d = 0.249 mm)	600 HV 30 (d =0.304 mm)	200 HV 30 (d = 0.527 mm)
Test force F	$\Delta F = 10^{-3} F$ ($\Delta HV/HV = \Delta F/F$)	0.000001	0.000001	0.000001
Indentation diagonal Length d	Δd = 1.1 μm ($\Delta HV/HV = -2\Delta d/d$)	0.0000781	0.0000524	0.0000174
Plane angle α	$\Delta\alpha$= 0.02º ($\Delta HV/HV = \Delta\alpha/2\tan(\alpha/2)$)	4×10^{-9}	4×10^{-9}	4×10^{-9}
Tip radius Δr	—	—	—	—
Length of line of	Δc = 0.6 μm	0.00001138	0.0000076	0.00000254

junction Δc	(ΔHV/HV = 1.4$\Delta c/d$)			
u^2 = (1/3)$\Sigma(\Delta HV_i/HV_i)^2$		0.00003016	0.00002033	0.00000698
$U_{rel} = 2(u^2)^{1/2}$		0.011	0.009	0.005
U_{rel}, %		1.1	0.9	0.5

Table B.1.4 Evaluation of the uncertainty for HV 10

Influencing Quantity x_i	Value a_i	Variances $(\Delta HV_i/HV_i)^2$		
		900 HV10 (d = 0.144 mm)	600HV10 (d = 0.176 mm)	200HV10 (d = 0.304 mm)
Test force F	$\Delta F = 10^{-3} F$ (ΔHV/HV= $\Delta F/F$)	0.000001	0.000001	0.000001
Indentation diagonal Length d	Δd = 0.7 μm (ΔHV/HV = -2$\Delta d/d$)	0.0000945	0.0000633	0.0000212
Plane angle α	$\Delta\alpha$= 0.02° (ΔHV/HV = $\Delta\alpha$/2tan(α/2))	4×10^{-9}	4×10^{-9}	4×10^{-9}
Tip radius Δr	—	—	—	—
Length of line of junction Δc	Δc = 0.6 μm (ΔHV/HV = 1.4$\Delta c/d$)	0.0000340	0.0000228	0.0000076
u^2 = (1/3)$\Sigma(\Delta HV_i/HV_i)^2$		0.0000432	0.0000290	0.00000993
$U_{rel} = 2(u^2)^{1/2}$		0.013	0.011	0.006
U_{rel}, %		1.3	1.1	0.6

Table B.1.5 Evaluation of the uncertainty for HV 1

Influencing Quantity x_i	Value a_i	Variances $(\Delta HV_i/HV_i)^2$		
		900 HV 1 (d = 0.0454 mm)	600 HV 1 (d = 0.0556 mm)	200 HV 1 (d = 0.0963 mm)
Test force F	$\Delta F = 1.4\times10^{-3} F$ (ΔHV/HV= $\Delta F/F$)	0.00000196	0.00000196	0.00000196
Indentation diagonal Length d	Δd = 0.4 μm (ΔHV/HV = -2$\Delta d/d$)	0.0003105	0.0002070	0.0000690
Plane angle α	$\Delta\alpha$= 0.01° (ΔHV/HV = $\Delta\alpha$/2tan(α/2))	0.0000001	0.0000001	0.0000001
Tip radius Δr	—	—	—	—
Length of line of junction Δc	Δc = 0.5 μm (ΔHV/HV = 1.4$\Delta c/d$)	0.0002377	0.0001585	0.0000528
u^2 = (1/3)$\Sigma(\Delta HV_i/HV_i)^2$		0.0001834	0.0001225	0.0000413
$U_{rel} = 2(u^2)^{1/2}$		0.027	0.022	0.013
U_{rel}, %		2.7	2.2	1.3

B.2 CMS / ITRI (Chinese Taipei)

B.2.1 Results of the verification

Results of the verification of standard hardness testing machine is shown in Table B.2.1.

Table B.2.1 Verification results

Item	Test force	Length measuring device	Angle between opposite faces of the intender (if possible)	Length of the line of conjunction at the point of the indenter (if possible)
Symbol	u_F [N]	u_l [μm]	α [degree]	c [μm]
Value	HV1: 9.33×10^{-3} HV10: 1.27×10^{-2} HV30: 6.65×10^{-2}	—	136°±0.1°	0.25

B.2.2 Uncertainty estimation

The uncertainty was estimated according to the ISO GUM. The following influence quantities have been considered:

(a) the test force
(b) the length measuring system
(c) the plane angle of the indenter
(d) the length of line of junction of the indenter
(e) tip radius
(f) numerical aperture
(g) parallax
(h) temperature

The expanded uncertainty (95% confidence level) are estimated and listed as below:

For HV1: $U = 4.0\times10^{-2}$ of H
For HV10: $U = 1.3\times10^{-2}$ of H
For HV30: $U = 1.3\times10^{-2}$ of H

B.3 SCL (Hong Kong, China)

B.3.1 Results of the verification

Results of the verification of standard hardness testing machine is shown in Table B.3.1.

Table B.3.1 Verification results

Item	Test force	Length measuring device	Angle between opposite faces of the intender (if possible)	Length of the line of conjunction at the point of the indenter (if possible)
Symbol	u_F [N]	u_l [μm]	α [degree]	c [μm]
Value	1.4 (Note 1) 1.6 (Note 2)	1.3 (Note 3)	136° 19´ & 136° 18´ (Note 4)	0.5 (Note 4)

Note 1: The expanded uncertainty of the test force is 1.4 N with a coverage factor of 2.04 at HV10 level.
Note 2: The expanded uncertainty of the test force is 1.6 N with a coverage factor of 2.04 at HV30 level.
Note 3: The expanded uncertainty of the indentation measuring device is 1.3 μm with a coverage factor of 2.00.
Note 4: According to the measurement results of Japan Bearing Inspection Institute.

B.4 NMIJ / AIST (Japan)

B.4.1 Results of the verification

Results of the verification of standard hardness testing machine is shown in Table B.4.1.

Table B.4.1 Verification results

Item	Test force	Length measuring device	Angle between opposite faces of the intender (if possible)	Length of the line of conjunction at the point of the indenter (if possible)
Symbol	u_F [N][a]	u_l [μm][b]	α [degree]	c [μm]
Value	HV1: 0.0536 HV10: 0.0853 HV30: 0.1595	10×: 0.644 20×: 0.422 40×: 0.437 100×: 0.394	136.10° 136.07°	0.036[c]

[a] Expanded uncertainty including the uncertainty of the force proving instruments.
[b] Expanded uncertainty including the uncertainty of reference standards. Human factors are not considered.

[c] Evaluated with an atomic force microscope.

B.3.2 Uncertainty estimation

The uncertainties are 6 HV, 16 HV and 24 HV for nominal measured values of 200 HV, 600 HV and 900 HV respectively.

B.4.2 Uncertainty estimation

An example of the uncertainty budget is shown in Table B.5.2.

Table B.5.2 Uncertainty budget for 200 HV 1 level.

Source x_i	Uncertainty $u(x_i)$	Sensitivity coefficient c_i	Uncertainty contribution, HV
Testing force F, N	0.0237	21.81[a]	0.516
Measuring system d, μm	0.222	-4.594[b]	1.021
Reproducibility of measurement d_{rep}, μm	0.433	-4.594[b]	1.990
Angle between opposite faces of indenter α, °	0.058	0.7541	0.044
Length of the line of conjunction of the point of indenter c, μm	0.29	0.01519	0.0044
Testing force application time t_a, s	0.58	0.0006625	0.00038
Testing force duration time t_F, s	0.58	0.06005	0.034
Nonuniformity of the reference block σ_H, HV	2.063	1	2.063
Combined uncertainty u_c, HV			3.087
Expanded uncertainty U (k = 2), HV			6.174
Relative expanded uncertainty U_{rel}			2.89 %

[a] The value for the hardness of 213.869 HV 1.

[b] The value for the diagonal length of 213.869 μm.

The results of uncertainty estimation at all hardness levels are summarized in Table B.4.3.

Table B.4.3 Summary of the uncertainty of measurement.

Hardness level	Average diagonal length, μm	Average hardness, HV	Expanded uncertainty, HV	Relative expanded uncertainty, %
200 HV 1	93.116	213.869	6.174	2.89
200 HV 10	294.985	213.106	2.390	1.12
200 HV 30	512.489	211.811	2.478	1.17
600 HV 1	55.427	603.612	28.439	4.71
600 HV 10	174.404	609.656	7.771	1.27
600 HV 30	302.592	607.577	8.133	1.34
900 HV 1	45.049	913.760	29.977	3.28
900 HV 10	142.893	908.190	14.776	1.63
900 HV 30	247.952	904.858	18.271	2.02

As seen in the uncertainty budget (Table B.4.2), it is found that the uncertainty of diagonal length measurement is the most significant uncertainty source. Fig. B.4.1 represents the relation between the diagonal length of indentation and the relative expanded uncertainty of hardness measurement.

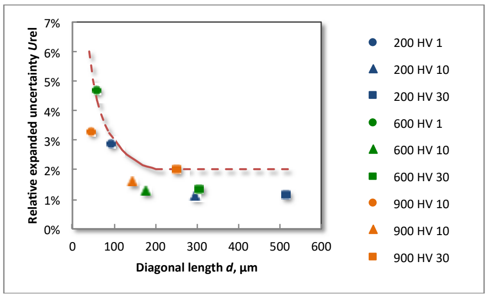

Fig. B.4.1 Relative expanded uncertainty of hardness measurement as the function of diagonal length of indentation.

In the chart, the dotted line represents claimed CMC of NMIJ, *i. e.*,

(1.0 + 200/d) % for d < 200 µm
2.0 % for d ≥ 200 µm,

where d is the diagonal length of indentation. The chart shows that the uncertainty of measurement at each hardness level in this comparison is smaller than the claimed CMC (It means that our CMC is on the safe side). The uncertainty reported with measurement results of this comparison is derived from the CMC.

B.5 KRISS (Korea)

B.5.1 Results of the verification

Results of the verification of standard hardness testing machine is shown in Table B.5.1.

Table B.5.1 Verification results

Item	Test force	Length measuring device	Angle between opposite faces of the intender (if possible)	Length of the line of conjunction at the point of the indenter (if possible)
Symbol	u_F [N]	u_l [µm]	α [degree]	c [µm]
Value	9.807 N : 0.08 % 98.07 N : 0.03 % 294.2 N : 0.023 %	0.16 % ~ 0.8 %	For 9.807 N : 135.97° For 98.07 N ~ 294.2 N : 135.98°	—

B.5.2 Uncertainty estimation

The uncertainty budgets are shown in Tables B.5.2 to B.5.10.

Table B.5.2 Uncertainty budget at 900 HV 1 level

Source	Estimated value	Unit	Uncertainty of item	Sensitivity coefficient	Uncertainty [HV]
Test force, u_F [N]	9.807	N	0.00785	93.994	0.74
Measuring device, u_l [µm]	44.85	µm	0.165	-41.106	-6.78
Angle between opposite faces of the indenter, α [°]	136	°	0.083	3.250	0.27
Line of conjunction of the indenter, c [µm]	1	µm	0.289	-29.066	-8.40
Uniformity of hardness [HV]	921.8	HV	0.666	1	0.67
Combined standard uncertainty					10.84
Expanded uncertainty (k = 2), HV					21.68
Relative Expanded uncertainty (k = 2), %					2.35

Table B.5.3 Uncertainty budget at 900 HV 10 level

Source	Estimated value	Unit	Uncertainty of item	Sensitivity coefficient	Uncertainty [HV]
Test force, u_F [N]	98.07	N	0.01426	9.137	0.13
Measuring device, u_l [μm]	143.9	μm	0.380	-12.458	-4.73
Angle between opposite faces of the indenter, *α* [°]	136	°	0.083	3.159	0.26
Line of conjunction of the indenter, *c* [μm]	2	μm	0.577	-8.809	-5.08
Uniformity of hardness [HV]	896.1	HV	2.216	1	2.22
Combined standard uncertainty					7.30
Expanded uncertainty (k = 2), HV					14.60
Relative Expanded uncertainty (k = 2), %					1.63

Table B.5.4 Uncertainty budget at 900 HV 30 level

Source	Estimated value	Unit	Uncertainty of item	Sensitivity coefficient	Uncertainty [HV]
Test force, u_F [N]	294.2	N	0.033744	3.049	0.10
Measuring device, u_l [μm]	249.3	μm	0.41	-7.202	-2.95
Angle between opposite faces of the indenter, *α* [°]	136	°	0.083	3.162	0.26
Line of conjunction of the indenter, *c* [μm]	2	μm	0.577	-5.093	-2.94
Uniformity of hardness [HV]	896.9	HV	1.387	1	1.39
Combined standard uncertainty					4.40
Expanded uncertainty (k = 2), HV					8.80
Relative Expanded uncertainty (k = 2), %					0.98

Table B.5.5 Uncertainty budget at 600 HV 1 level

Source	Estimated value	Unit	Uncertainty of item	Sensitivity coefficient	Uncertainty [HV]
Test force, u_F [N]	9.807	N	0.00785	62.629	0.49
Measuring device, u_l [μm]	54.95	μm	0.165	-22.355	-3.69
Angle between opposite faces of the indenter, *α* [°]	136	°	0.083	2.166	0.18
Line of conjunction of the indenter, *c* [μm]	1	μm	0.289	-15.793	-4.56
Uniformity of hardness [HV]	614.2	HV	1.058	1	1.06
Combined standard uncertainty					5.99
Expanded uncertainty (k = 2), HV					11.97
Relative Expanded uncertainty (k = 2), %					1.95

Table B.5.6 Uncertainty budget at 600 HV 10 level

Source	Estimated value	Unit	Uncertainty of item	Sensitivity coefficient	Uncertainty [HV]
Test force, u_F [N]	98.07	N	0.01426	6.118	0.09
Measuring device, u_l [μm]	175.8	μm	0.38	-6.826	-2.59
Angle between opposite faces of the indenter, *α* [°]	136	°	0.083	2.115	0.18
Line of conjunction of the indenter, *c* [μm]	2	μm	0.577	-4.827	-2.79
Uniformity of hardness [HV]	600	HV	0.212	1	0.21
Combined standard uncertainty					3.82
Expanded uncertainty (k = 2), HV					7.64

Relative Expanded uncertainty (k = 2), %	1.27

Table B.5.7 Uncertainty budget at 600 HV 30 level

Source	Estimated value	Unit	Uncertainty of item	Sensitivity coefficient	Uncertainty [HV]
Test force, u_F [N]	294.2	N	0.033744	2.043	0.07
Measuring device, u_l [μm]	304.7	μm	0.43	-3.952	-1.70
Angle between opposite faces of the indenter, α [°]	136	°	0.083	2.119	0.18
Line of conjunction of the indenter, c [μm]	2	μm	0.577	-2.794	-1.61
Uniformity of hardness [HV]	601.1	HV	0.396	1	0.40
Combined standard uncertainty					2.38
Expanded uncertainty (k = 2), HV					4.77
Relative Expanded uncertainty (k = 2), %					0.79

Table B.5.8 Uncertainty budget at 200 HV 1 level

Source	Estimated value	Unit	Uncertainty of item	Sensitivity coefficient	Uncertainty [HV]
Test force, u_F [N]	9.807	N	0.00785	21.984	0.17
Measuring device, u_l [μm]	92.73	μm	0.13	-4.656	-0.60
Angle between opposite faces of the indenter, α [°]	136	°	0.083	0.760	0.06
Line of conjunction of the indenter, c [μm]	1	μm	0.289	-3.282	-0.95
Uniformity of hardness [HV]	215.6	HV	0.703	1	0.70
Combined standard uncertainty					1.34
Expanded uncertainty (k = 2), HV					2.68
Relative Expanded uncertainty (k = 2), %					1.24

Table B.5.9 Uncertainty budget at 200 HV 10 level

Source	Estimated value	Unit	Uncertainty of item	Sensitivity coefficient	Uncertainty [HV]
Test force, u_F [N]	98.07	N	0.01426	2.162	0.03
Measuring device, u_l [μm]	295.76	μm	0.39	-1.434	-0.56
Angle between opposite faces of the indenter, α [°]	136	°	0.083	0.747	0.06
Line of conjunction of the indenter, c [μm]	2	μm	0.577	-1.014	-0.59
Uniformity of hardness [HV]	212	HV	0.26	1	0.26
Combined standard uncertainty					0.85
Expanded uncertainty (k = 2), HV					1.71
Relative Expanded uncertainty (k = 2), %					0.80

Table B.5.10 Uncertainty budget at 200 HV 30 level

Source	Estimated value	Unit	Uncertainty of item	Sensitivity coefficient	Uncertainty [HV]
Test force, u_F [N]	294.2	N	0.033744	0.722	0.02
Measuring device, u_l [μm]	511.86	μm	0.41	-0.830	-0.34
Angle between opposite faces of the indenter, α [°]	136	°	0.083	0.749	0.06
Line of conjunction of the indenter, c [μm]	2	μm	0.577	-0.586	-0.34
Uniformity of hardness [HV]	212.3	HV	0.243	1	0.24

Combined standard uncertainty	0.54
Expanded uncertainty (k = 2), HV	1.08
Relative Expanded uncertainty (k = 2), %	0.51

B.6 NIMT (Thailand)

B.6.1 Results of the verification

Results of the verification of standard hardness testing machine is shown in Table B.6.1.

Table B.6.1 Verification results

Item	Test force	Length measuring device	Angle between opposite faces of the intender (if possible)	Length of the line of conjunction at the point of the indenter (if possible)
Symbol	u_F [N]	u_l [μm]	α [degree]	c [μm]
Value	0.1 %	0.5 % for d ≥ 160 μm 0.8 μm for d < 169 μm	136° 2′ ± 1′ for S/N 5079 135° 58′ - 135° 59′ for S/N TDS41777	0.37 μm ± 0.02 μm for S/N 5079 0.5 μm for S/N TDS41777

Remarks:
Indenter S/N 5079 was used for test force ≤ 1 kgf.
Indenter S/N TDS41777 was used for test force > 1 kgf.

B.6.2 Uncertainty budgets

B.6.2.1 Uncertainty of test force

Test force 10 and 30 kgf were calibrated with load cell class 00. They are traceable to SI unit through JCSS certificate.

Test force 1kgf was calibrated by using weight 1 kg and balance. It is traceable to SI unit through DKD certificate.

Table B.6.2 Uncertainty of force proving instruments.

Test force kgf	Error (%)	U_e (%)
1	0.001	0.05
10	-0.017	0.05
30	-0.031	0.05

The tolerance of force (0.1%) was used as the expanded uncertainty of test force. The normal distribution should be assumed.

B.6.2.2 Uncertainty of the length measuring device

The microscope, Olympus model: BH2-UMA, and Akashi HM124 were adapted with digital camera high resolution 4 Mpixles and 6 Mplixles, respectively. Both were calibrated by glass scale. The calibration result was within 0.45 μm. Diagonal length had to be measured by using both microscopes. Agreement value of 0.5 % between 2 microscope was used for confirming the accuracy of the measurement. The accuracy was equal to 0.8 μm if the diagonal length was less than 160 μm and was equal to 0.5% of reading if the diagonal length was greater then 160 μm.

Table B.6.3 Accuracy of diagonal length meareument

Diagonal length	Accuracy
< 160 μm	0.8 μm
≥ 160 μm	0.5 %rdg

B.6.2.3 Uncertainty of the plane angle of the indenter and the length of line of junction of the indenter

NIMT used 2 indenters for test force 1 kgf and test force more than 10 kgf. And they were calibrated by MPA (DKD laboratory) and JBI, respectively. Both indenters conformed to ISO 6507-3 and its tolerance shuold be used as expanded uncertainty as below.

Table B.6.4 Tolerance of the geometry of indenters.

Indenter	Requirement	Measured Value	U_e	Expected Value
No.5079	plane angle of the indenter.	136° 2′	±1′	136°±6′
for ≤ 1kgf	length of line of junction of the indenter	0.00037mm	0.00002 mm	0.001 mm
TDS41777	plane angle of the indenter.	135° 58′−59′	—	136°±6′
for > 1kgf	length of line of junction of the indenter	>0.0005mm	—	0.001 mm

B.6.2.4 Uncertainty of testing cycle

Testing cycle of measurement was maintained under the following conditions:

Loading speed 60 μm/s ± 1 μm/s for test force > 1 kgf
Time for application of the test force < 1 s for test force ≤ 1 kgf
Duration of application of the test force 14 s ± 0.5 s

From these MPE of testing cycle, it was estimated to effect to the hardness measurement with ±1% at 2σ with normal distribution.

B.6.2.5 Uncertainty budgets

Table B.6.5 Example 1, d = 40 μm

Quantity x_i	Uncertainty	Standard Uncertainty $u(x_i)$	Probability Distribution	Sensitivity Coefficient c_i	Uncertainty Contribution $u(y_i)$
test force (%)	0.1	0.05	Normal	1	0.05
diagonal (%) Note1	2	1	Normal	2	2.00
angle (°)	0.1	0.058	Rectangular	0.3526	0.02
line (%) Note2	2.5	1.443	Rectangular	1.4142	2.04
testing cycle (%)	1	0.58	Rectangular	1	0.58
Uniformity (%)	0.5	0.25	Normal	1	0.25
u_c					2.93
U_e					5.85

Note1 Diagonal length <160μm $u(d)$ = 0.8 μm/d %
Note2 d = 100 μm $u(l)$ = (1/100)%

Table B.6.6 Example 2, d = 100 μm

Quantity x_i	Uncertainty	Standard Uncertainty $u(x_i)$	Probability Distribution	Sensitivity Coefficient c_i	Uncertainty Contribution $u(y_i)$
test force (%)	0.1	0.05	Normal	1	0.05

diagonal (%) Note1	0.8	0.4	Normal	2	0.80
angle (°)	0.1	0.058	Rectangular	0.3526	0.02
line (%) Note2	1	0.577	Rectangular	1.4142	0.82
testing cycle (%)	1	0.58	Rectangular	1	0.58
Uniformity (%)	0.5	0.25	Normal	1	0.25
u_c					1.31
U_e					2.61

Note1 Diagonal length < 160 μm $u(d)$ = 0.8 μm/d %

Note2 d = 100 μm $u(l)$= (1/100)%

Table B.6.7 Example 3, d = 200 μm

Quantity x_i	Uncertainty	Standard Uncertainty $u(x_i)$	Probability Distribution	Sensitivity Coefficient c_i	Uncertainty Contribution $u(y_i)$
test force (%)	0.1	0.05	Normal	1	0.05
diagonal (%) Note1	0.5	0.25	Normal	2	0.50
angle (°)	0.1	0.058	Rectangular	0.3526	0.02
line (%) Note2	0.5	0.289	Rectangular	1.4142	0.41
testing cycle (%)	1	0.58	Rectangular	1	0.58
Uniformity (%)	0.5	0.25	Normal	1	0.25
u_c					0.90
U_e					1.81

Note1 Diagonal length ≥ 160 μm $u(d)$=0.5 %

Note2 d = 200 μm $u(l)$= (1/200)%

Table B.6.8 Example 3, d = 400 μm

Quantity x_i	Uncertainty	Standard Uncertainty $u(x_i)$	Probability Distribution	Sensitivity Coefficient c_i	Uncertainty Contribution $u(y_i)$
test force (%)	0.1	0.05	Normal	1	0.050
diagonal (%) Note1	0.5	0.25	Normal	2	0.500
angle (°)	0.1	0.058	Rectangular	0.3526	0.020
line (%) Note2	0.25	0.144	Rectangular	1.4142	0.204
testing cycle (%)	1	0.58	Rectangular	1	0.577
Uniformity (%)	0.5	0.25	Normal	1	0.250
u_c					0.83
U_e					1.66

Note1 Diagonal length ≥160μm $u(d)$=0.5 %

Note2 d = 400 μm $u(l)$= (1/400)%

Table B.6.9 Example 3, d = 550 μm

Quantity x_i	Uncertainty	Standard Uncertainty $u(x_i)$	Probability Distribution	Sensitivity Coefficient c_i	Uncertainty Contribution $u(y_i)$
test force (%)	0.1	0.05	Normal	1	0.050
diagonal (%) Note1	0.5	0.25	Normal	2	0.500
angle (°)	0.1	0.058	Rectangular	0.3526	0.020
line (%) Note2	0.18	0.105	Rectangular	1.4142	0.148
testing cycle (%)	1	0.58	Rectangular	1	0.577
Uniformity (%)	0.5	0.25	Normal	1	0.250
u_c					0.82
U_e					1.64

Note1 Diagonal length ≥160μm $u(d)$=0.5 %

Note2 d = 550 μm $u(l)$= (1/550)%

B.6.2.6 Uncertainty in Vickers hardness measurement

1 + 200/*d* % for d < 200 μm
2 % for *d* ≥ 200 μm

B.7 VMI (Vietnam)

B.7.1 Results of the verification

Results of the verification of standard hardness testing machine is shown in Table B.7.1.

Table B.7.1 Verification results

Item	Test force	Length measuring device	Angle between opposite faces of the intender (if possible)	Length of the line of conjunction at the point of the indenter (if possible)
Symbol	u_F [N]	u_l [μm]	α [degree]	c [μm]
Value	0.1	0.1	136°	0.5