HARDMEKO 2004
Hardness Measurements Theory and Application in Laboratories and Industries
11-12 November, 2004, Washington, D.C.,USA

CCM VICKERS KEY COMPARISON - STATE OF THE ART AND PERSPECTIVES

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Abstract - In the framework of the Working Group on Hardness (WGH) of the Consultative Committee for Mechanical Measurements (CCM) in the year 2003 the key comparison Vickers was finished. In the comparison the hardness laboratories of 10 national metrology institutes participated: IMGC (Italy), NIST (USA), INMETRO (Brazil), NIM (P.R. China), KRISS (Republic of Korea), NMIJ (Japan), CMI (Czech Republic), GUM (Poland), NPL (U.K.), and PTB (Germany) served as the pilot laboratory. The comparison of the Vickers primary hardness standard machines was carried out with three sets of hardness reference blocks of the Vickers scales HV0,2, HV1 and HV 30 each with the hardness levels 240 HV, 540 HV and 840 HV.

The Vickers key comparison for all used test forces ranges (Micro Vickers, Small Force Vickers, Macro Vickers) delivered valuable metrological data, and its main results are presented. The ways to determine the reference values are discussed. The uncertainties of the reference values can be considered as the present accuracy limits of Vickers measurements in the investigated ranges of hardness scales.

Keywords: Vickers hardness, key comparison, measurement uncertainty, reference value

1 INTRODUCTION

On decision of the Consultative Committee for Mechanical Measurements of the CIPM its Working Group on Hardness conducted the key comparison Vickers in the time from March 2001 until March 2003. In the comparison participated the following national metrology institutes: IMGC (Italy), NIST (USA), INMETRO (Brazil), NIM (P.R. China), KRISS (Republic of Korea), NMIJ (Japan), CMI (Czech Republic), GUM (Poland), NPL (U.K.), and PTB (Germany) served as the pilot laboratory. In the comparison three sets of hardness reference blocks for the Vickers hardness scales HV0,2, HV1 and HV30 consisting each of three hardness reference blocks with the hardness levels 240 HV, 540 HV and 840 HV (that is altogether nine blocks) were used. The dimensions of the blocks are length 60 mm, width 60 mm, thickness 10 mm. The upper side of the blocks which is the measurement surface is finished. The blocks are manufactured as commercial products by Buderus Co., Germany.

2 MEASUREMENT RESULTS

The measurands used in this comparison were of two kinds. Firstly, the mean value each of eight hardness measurements on a hardness reference block had to be determined. The hardness measurements were made in the hardness scales HV0,2, HV1 and HV30 each for the nominal hardness levels 240 HV, 540 HV and 840 HV. The procedure of the hardness measurement is defined in ISO 6507-1 and -3. [1][2] Secondly, the mean diagonal length of nine reference indents had to be determined according to ISO 6507-1 and -

3. The reference indents represent indents for the hardness levels 240 HV, 540 HV and 840 HV, each for the Vickers scales HV0,2, HV1 and HV30.

As guideline for the estimation of the measurement uncertainty a draft guideline elaborated in the framework of the European Cooperation for Accrediation was used.[3]

The Fig. 1 until 3 depict the results of the comparison received for the hardness levels 240 HV, 540 HV and 840 HV.

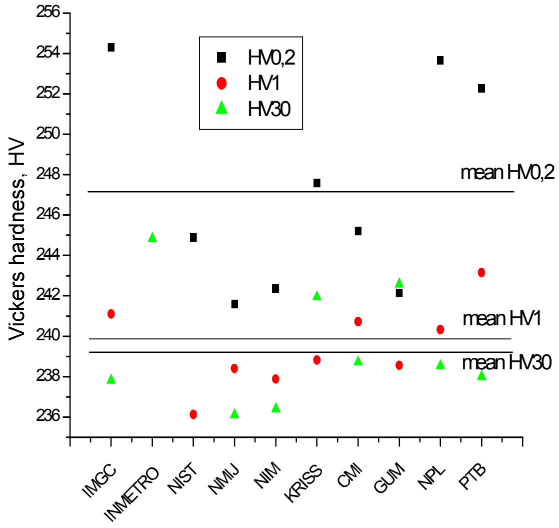

Fig. 1. Measurement results for the hardness level 240 HV

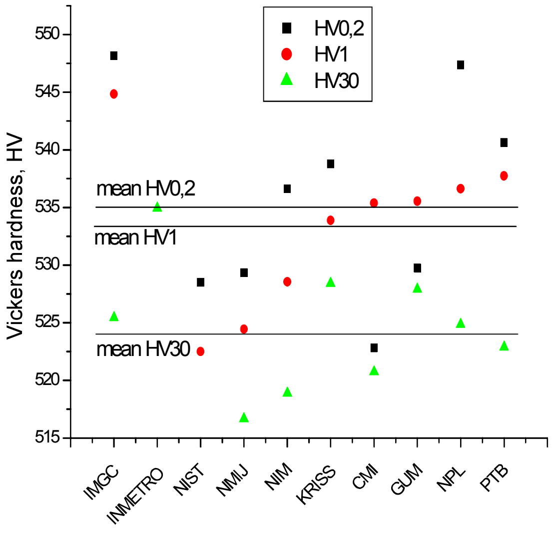

Fig. 2. Measurement results for the hardness level 540 HV

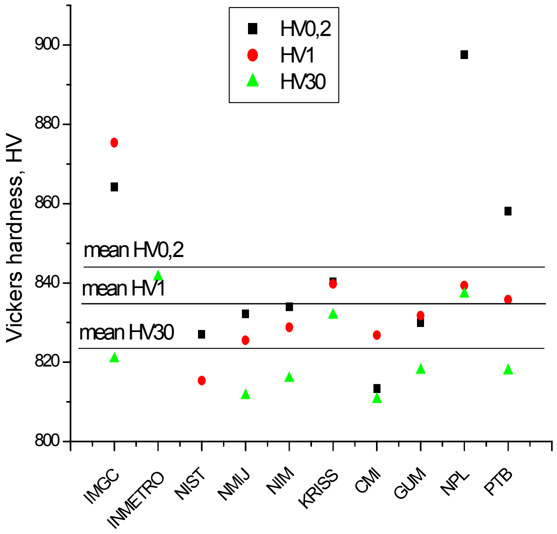

Fig. 3. Measurement results for the hardness level 840 HV

Fig. 4 represents the relative reproducibility s_{Inst} over the hardness.

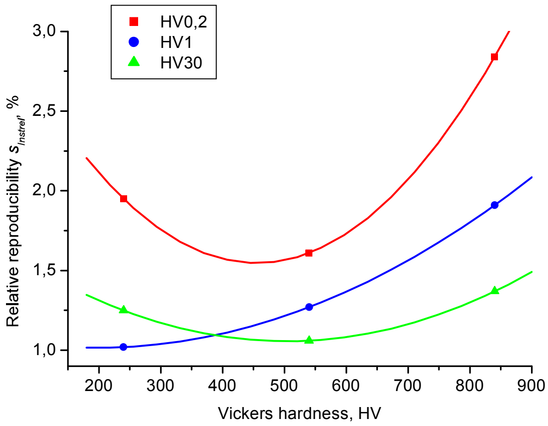

Fig. 4. Relative reproducibility $s_{Instrel}$ over Vickers hardness for the three scales HV0,2, HV1 and HV30 (fitted curves)

Fig. 4 illustrates the presently achievable degree of agreement for Vickers measurements in the scales HV0,2, HV1 and HV30 carried out by the participating national metrology institutes. Whereas for the high hardness level of 840 HV the uncertainty (expressed by the reproducibility) increases with decreasing test force, for the low hardness level of 240 HV the influence from the diagonal measurements becomes more evident. This may be the reason that $s_{Instrel}$ at 240 HV for HV30 is higher than for HV1.

3 DETERMINATION OF REFERENCE VALUES

For the calculation of the reference values two ways were tried:

1) Reference values based on the arithmetic mean
2) Reference values based on the weighted mean

In the following these two evaluation methods are compared with each other.

3.1 Reference values based on the arithmetic mean

The arithmetic mean value is calculated as follows:

$$\bar{x} = \frac{1}{n}\sum_{i-1}^{n} x_i \qquad (1)$$

with

$x_1, x_2,...,x_n$ = measurement results of n participants

n = number of participants

The uncertainty of the arithmetic mean value is expressed by the confidence interval:

$$u_{\bar{x}} = \frac{t \cdot s}{\sqrt{n}} \qquad (2)$$

with

t = factor of Student distribution ($t = \mathrm{f}(\alpha,n)$)

s = standard deviation of the measurement results of n participants

Finally, the coefficient E_n, which evaluates the agreement between the measurement deviations found in the comparison and the uncertainties stated by the participants, is calculated with the following expression:

$$E_n = \frac{x_{lab} - x_{ref}}{\sqrt{U_{lab}^2 + U_{ref}^2}} \qquad (3)$$

where x_{lab} is the measurement result of the participating laboratory, x_{ref} - the reference value, U_{lab} - the uncertainty stated by the participating laboratory and U_{ref} - the uncertainty of x_{ref}.

For the reference values only the measurement results with $E_n \leq 1$ are considered.

In the key comparison the following references values were received.

Table 1. Reference values and their uncertainties for the hardness measurements (evaluation by the participants)

Hardness scale	**Ref. value x_{ref}**	**Uncertainty of reference value U_{ref}**	
		Absolute unc., HV	**Relative unc., %**
240 HV0,2	247,09	4,57	1,85
240 HV1	239,46	1,70	0,71
240 HV30	239,44	2,44	1,02
540 HV0,2	535,78	7,17	1,34
540 HV1	533,28	6,25	1,17
540 HV30	524,53	4,52	0,86
840 HV0,2	844,07	20,69	2,45
840 HV1	835,40	13,71	1,64
840 HV30	822,83	9,17	1,11

3.2 Reference values based on the weighted mean

The reference values are calculated as the weighted mean based on the uncertainties of the participants. The weighted mean is calculated as follows:

$$\bar{x}_w = \frac{x_1 u_1^{-2} + x_2 u_2^{-2} + \ldots + x_n u_n^{-2}}{u_1^{-2} + u_2^{-2} + \ldots + u_n^{-2}} \qquad (4)$$

with

$x_1, x_2,...,x_n$ = measurement results of n participants

$u_1, u_2,...,u_n$ = measurement uncertainties of n participants

The uncertainty of the weighted mean according to the law of error propagation follows from:

$$U_{xw} = \sqrt{(u_1 S)^{-2} + (u_2 S)^{-2} + \ldots + (u_n S)^{-2}} \qquad (5)$$

with

$$S = u_1^{-2} + u_2^{-2} + \ldots + u_n^{-2}$$

The following references values based on the weighted mean were received.

Table 2. Reference values and their uncertainties for the hardness measurements (evaluation by the participants)

Hardness scale	**Ref. value x_{ref}**	**Uncertainty of reference value U_{ref}**	
		Absolute unc., HV	**Relative unc., %**
240 HV0,2	246,00	2,54	0,45
240 HV1	238,48	1,37	0,42
240 HV30	238,58	0,54	0,23
540 HV0,2	531,29	6,65	1,13
540 HV1	534,89	4,36	0,82
540 HV30	524,97	1,39	0,26
840 HV0,2	827,76	11,86	1,36
840 HV1	828,36	6,86	0,83
840 HV30	813,48	3,04	0,37

The comparison of the uncertainties given in the tables 1 and 2 reveals that the calculation based on the weighted mean delivers smaller uncertainties of the reference values than the corresponding calculation based on the arithmetic mean. The reason is the property of the uncertainty of the weighted mean that it is smaller than the smallest uncertainty of any measurement result contributing to the weighted mean.

Fig. 5 depicts the dependency of the uncertainty on the hardness level and the test force (resp. hardness scale).

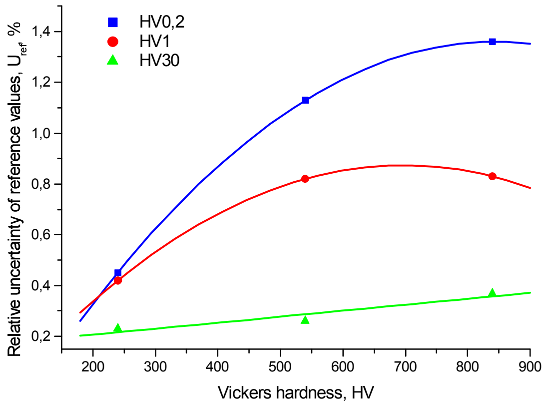

Fig. 5. Dependency of the relative uncertainty of the reference values on the hardness level and the test force (resp. hardness scale)

Fig. 5 shows that the relative uncertainty of the reference values – as expected – increases with the hardness, whereby smaller test forces deliver higher uncertainties than larger test forces. This reflects the fact that the Vickers hardness uncertainty is strongly influenced by the uncertainty of the diagonal measurement.

3 ESTIMATION OF THE UNCERTAINTY BASED ON A UNIFIED METHOD

The application of the EA draft "Guideline to the estimation of the uncertainty of the Brinell and the Vickers measuring method" already guaranteed a rather uniform approach of all participants to the estimation of the measurement uncertainty. Nevertheless, the amount of considered influence quantities was different, and the type of distribution was also not always considered. So the individual application of this guideline in several cases led to uncertainty values which are not sufficiently comparable.

Therefore a unified estimation of the measurement uncertainty was made on the following basis.

Five influencing quantities are included in the calculation:

- Test force *F*
- Diagonal length *d*
- Pyramid angle of the indenter *α*
- Tip radius of the indenter *r*
- Length of indenter’s line of junction *c*

From the influencing quantities X_i measurement deviations Δx_i and uncertainties in the form of standard deviation s_i (type A) and a_i (type B) are considered.

Standard uncertainty:

$$u^2(x_i) = s_i^2 \vee \frac{a_i^2}{3} \qquad (6)$$

Sensitivity coefficients:

$$\begin{aligned}
c_1 &= \frac{\partial HV}{\partial F} = \frac{0{,}204 \cdot \sin\alpha/2}{d^2} \\
c_2 &= \frac{\partial HV}{\partial d} = \frac{-0{,}408 \cdot F \cdot \sin\alpha/2}{d^3} \\
c_3 &= \frac{\partial HV}{\partial \alpha} = \frac{0{,}102 \cdot F \cdot \cos\alpha/2}{d^2} \\
c_4 &= \frac{\partial HV}{\partial r} = \frac{-0{,}204 \cdot F \cdot \sin\alpha/2}{d^3} \cdot (1{,}099 + 1{,}1515 \cdot \frac{r}{d}) \\
c_5 &= \frac{\partial HV}{\partial c} = -\frac{0{,}2856 \cdot F \cdot \sin\alpha/2}{d^3}
\end{aligned} \qquad (7)$$

Single hardness deviation:

$$\Delta H_i = c_i \cdot \Delta x_i \qquad (8)$$

Variances:

$$u^2(y_i) = c_i^2 u^2(x_i) \tag{9}$$

Combined standard uncertainty:

$$u(H) = \sqrt{\sum_{i=1}^{n} u^2(y_i)} \tag{10}$$

Sum of hardness deviations:

$$\Delta H = \sum_{i=1}^{n} \Delta H_i \tag{11}$$

Effective degrees of freedom, according to the Welch-Satterthwaite formula:

$$\nu_{eff} = \frac{u^4(y)}{\sum_{i=1}^{\nu} \frac{u_i^4(y)}{\nu_i}} \tag{12}$$

Coverage factor:

$$k = f(\nu_{eff}, P) \tag{13}$$

Expanded standard uncertainty:

$$U(H) = k \cdot u(H) + |\Delta H| \tag{14}$$

Relative expanded standard uncertainty:

$$U_{rel}(H) = \frac{U(H)}{H} \cdot 100, \% \tag{15}$$

According to this unified procedure for the estimation of measurement uncertainty, the following measurement uncertainties for the participants were received. If the participants had omitted the indenter tip radius because they did not have the facility to measure it, a value of r = 0,5 µm with an uncertainty of 0,2 µm was set in.

Fig. 6 summarizes the relative measurement uncertainty obtained by the unified method for all partners in the comparison and for all hardness scales.

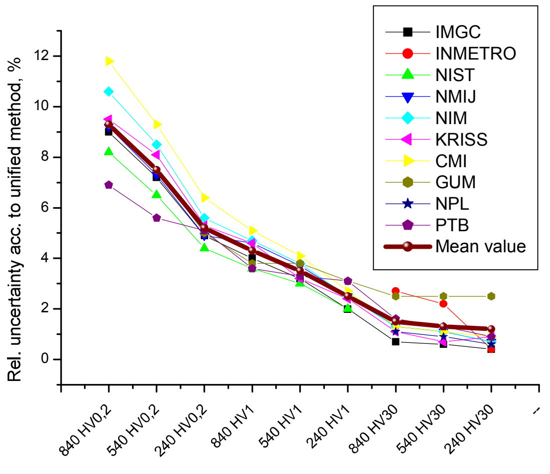

Fig. 6. Relative measurement uncertainty obtained by the unified method for all partners and all hardness scales

Fig. 6 clarifies that the measurement uncertainty mainly depends on the uncertainty of diagonal length measurement, because the diagonal length in the given order of the hardness scales is increasing. Whereas the variation of the uncertainty for the scale 840 HV0,2 is 4,9 %, this variation for the scale 240 HV30 reduces to 2,9 % for the participating laboratories.

The question whether the measurement deviations found in the comparison can be explained by the uncertainties stated by the participants is evaluated with the coefficient E_n.

Fig. 7 presents the E_n values received for the unified method of evaluating the measurement uncertainty where the reference values were calculated using the arithmetic mean.

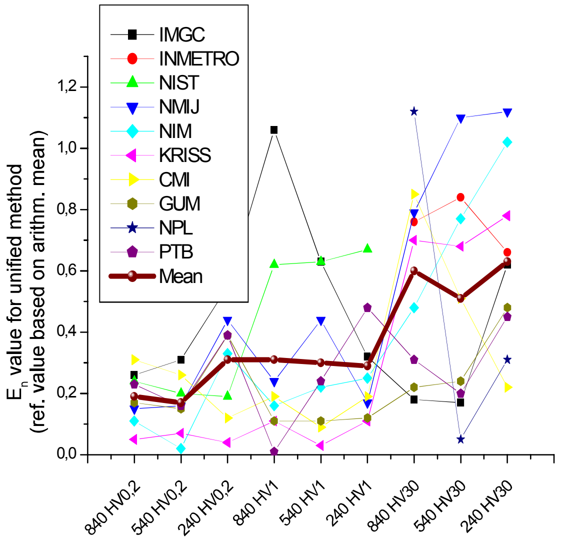

Fig. 7. E_n values for the unified method of evaluating the

measurement uncertainty (reference values calculated using the arithmetic mean)

Fig. 7 displays for the mean value the rather clear tendency that the E_n value increases with increasing diagonal length (the hardness scales are here given in the order of increasing diagonal length). This means that the measurement uncertainty for larger diagonal lengths is underestimated. This underlines that the calibration methods for the length measuring system for large diagonal lengths should be improved. For only 5 from altogether 75 measurement results the limit $E_n \leq 1$ is exceeded.

In Fig. 8 the E_n values received for the arithmetic and the weighted mean are compared with each other.

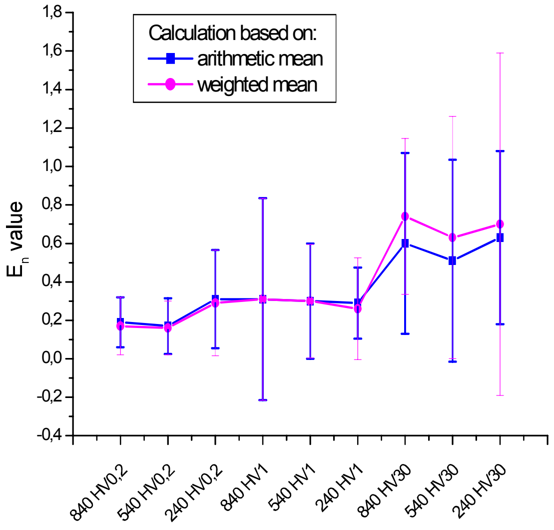

Fig. 8. E_n values received for the arithmetic and the weighted mean based on the unified method for the estimation of the measurement uncertainty

From Fig. 8 it is clear that the weighted mean for the large indentations obtained with the hardness scale HV30 deliver larger E_n values and larger variation. The reason for the larger variation is the propensity of the weighted mean to overestimate single results and therefore to distort the mean value if the number of single measurement results is small, as in the case of this comparison ($n = 10$).

Moreover, the E_n values derived from the arithmetic and from the weighted mean yield the result that the E_n values based on the arithmetic mean deliver less exceedings of the limit $E_n \leq 1$ than the E_n values based on the weighted mean. **This clarifies that the arithmetic mean is better suited for a small number of participants ($n = 10$).**

On the basis of the measurement uncertainties according to the unified procedure based on the arithmetic mean the reference values were recalculated. Measurement results with $E_n > 1$ have been excluded. The results are given in the following Table 3.

Table 3. Reference values and their uncertainties for the hardness measurements (evaluation by the unified procedure based on the arithmetic mean)

Hardness scale	**Reference value x_{ref}, HV**	**Difference reference values (part.-unif.)**		**Uncertainty of reference value U_{ref}**	
		abs., HV	**Rel., %**	**Abs. unc., HV**	**Rel. unc., %**
240HV0,2	247,09	0,00	0,00	4,17	1,69
240HV1	239,46	0,00	0,00	1,70	0,71
240HV30	240,35	-0,91	-0,38	2,24	0,93
540HV0,2	535,78	0,00	0,00	7,17	1,34
540HV1	533,28	0,00	0,00	5,70	1,07
540HV30	525,51	-0,98	-0,19	4,10	0,78
840HV0,2	844,07	0,00	0,00	21,22	2,51
840HV1	830,40	5,00	0,60	6,80	0,82
840HV30	821,04	1,79	0,22	8,61	1,05

5 INFLUENCE OF THE LENGTH MEASURING SYSTEM

Because at Vickers measurements the length measuring deviation significantly influences the uncertainty of measurements, in the technical protocol the measurement of reference indents was included.

Fig. 9 shows the relative reproducibility of the diagonal measurement expressed by the standard deviation $s_{Instrel}$ over the hardness.

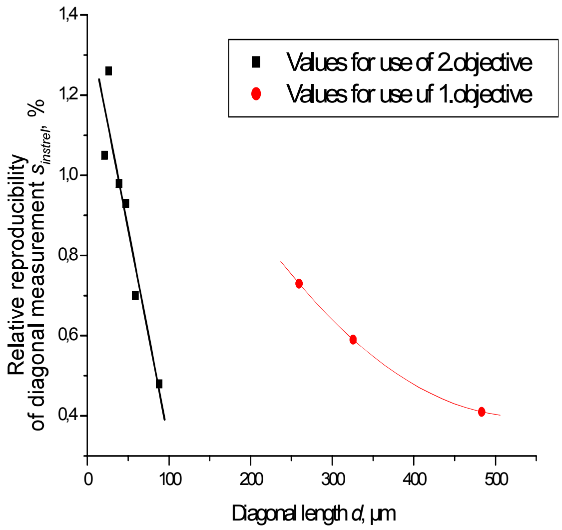

Fig. 9. Relative reproducibility of the diagonal measurement expressed by the standard deviation $s_{Instrel}$ over the diagonal length.

In Fig. 9 two groups of data attract the attention. These two groups of data correspond to two different objectives used for the diagonal measurement, one with higher magnification for smaller indents with diagonals $d < 100$ µm and the other with smaller magnification for indents with diagonals 250 µm $< d <$ 500 µm. For both groups the relative reproducibility $s_{instrel}$ decreases with increasing diagonal length d. But for the smaller diagonals the reproducibility increases much more sharply with decreasing diagonal length than for the larger diagonals. For the smaller diagonals ($d <$ 100 µm) the relative reproducibility is in a range $s_{instrel}$ = 0,5 % to 1,3 % and for the larger diagonals in a range $s_{instrel}$ = 0,4 % to 0,7 %.

6 INFLUENCE OF THE NUMERICAL APERTURE OF THE OBJECTIVE

Depending on the used equipment the participants have used optical length measuring systems with different magnifications and numerical apertures. The largest difference of numerical aperture ΔNA_{max} between the participants for the different indents was between 0,58 and 0,8. Therefore one can assume that the numerical aperture of the used objectives can influence the results of the length measurement.

In order to investigate this influence, partner IMGC measured the reference indents with a microscope to which belongs a set of five objectives. These objectives have the following magnifications and numerical apertures:

Magnification	32x	20x	10x	5x	2,5x
Numerical aperture	0,5	0,4	0,2	0,09	0,07

The result of the measurement of the reference indents with the 5 different objectives is shown in Fig. 10 as the relative length measuring deviations in dependence on the diagonal length and the numerical aperture.

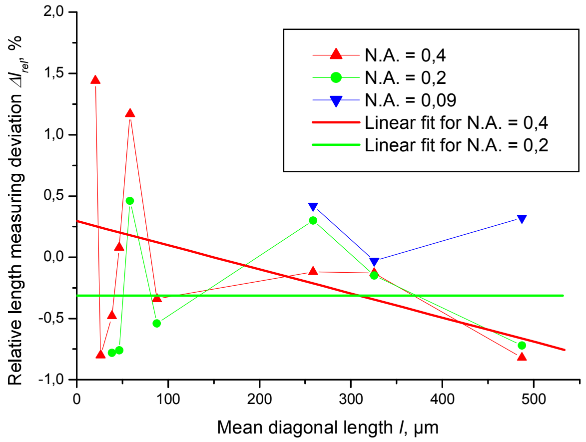

Fig. 10. Relative length measuring deviations in dependence on the diagonal length and the numerical aperture (for $N.A.$ = 0,5 the length measurement deviations are set = 0)

Fig. 10 clarifies that the effect of numerical aperture does not have a well defined slope and sign. Changing the objectives (and the numerical aperture), the measurements change very much (about ± 1 %), but in a random way.

From this analysis it is not possible to correct for the effect of the numerical aperture. One can only conclude that variations of the diagonal length of ±1 % can be justified, if different objectives and numerical apertures are used.

In future the investigation of the influence of the numerical aperture of the objective on the hardness deviation should be continued.

7 CONCLUSIONS

The CCM Vickers key comparison can be considered as a successful metrological exercise. Representatively it delivered for all three ranges of the test forces (Micro-Vickers, Small Force Vickers and Macro-Vickers scales) valuable metrological data. At present Vickers hardness reference blocks with high time-dependent stability and high local homogeneity, including high surface quality are available.

The uncertainties of the reference values are obviously the smallest uncertainties reached in the field of Vickers measurements worldwide so far. These uncertainties can be interpreted as the present limits of Vickers measurements in the investigated range of hardness scales. This is one important outcome of this Vickers key comparison.

In order to overcome these metrological limits in the future, it is recommended to concentrate metrological investigations on the following topics:

1) It was found that the calibration methods for the diagonal measurements, especially for diagonal lengths $d > 100$ µm should be improved.

2) The calibration methods for the parameters of the indenter geometry, like tip radius and length of the line of junction should be further developed.

3) The inputs from the participants to the used draft guideline for the estimation of the measurement uncertainty should be used for its further qualification. In this context it seems to be necessary to carry out experimental investigations on the determination of sensitivity coefficients for material dependent influences on the Vickers hardness measurement, especially influences of the force-time regime on the Vickers hardness.

4) For the further reduction of the uncertainty of Micro-Vickers measurements it is necessary to correct for the indenter deviations and to provide indenters with higher quality.

5) With the development of automatic Vickers indent measurements on the basis of CCD technique it is necessary to guarantee a high agreement of measurements both with CCD systems and with optical microscopes. This requires investigations on the influences of both methods on the hardness values.

6) For the diagonal measurements with optical microscopes the properties of the used optical system should be further investigated.

REFERENCES

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[3] EA Working group Hardness; Draft: Guideline to the estimation of the uncertainty of the Brinell and the Vickers measuring method, July 2002

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