### REPORT OF THE WORKING GROUP ON HARDNESS (WGH) ACTIVITIES (2005)

Prepared by A. Germak (Chairman) and S. Low (Secretary)

The Working Group on Hardness held its sixth meeting at the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland, USA on 15-16 November 2004. The meeting was scheduled to coincide with the international conference HARDMEKO 2004, held the previous week in Washington, DC.

#### Membership of the WGH

(\* Present at the last meeting)

- \* Edward G. Aslanyan (VNIIFTRI) Russia
- \* Gun Woong Bahng (KRISS) Korea Natalia G. Domostroyeva (VNIIM) Russia Cihan Kuzu (UME) Turkey
- \* Alessandro Germak (IMGC) Italy
- \* Konrad Herrmann (PTB) Germany
- \* Koichiro Hattori (NMIJ) Japan Anna Osinska Karczmarek (GUM) Poland
- \* Andy Knott (NPL) United Kingdom Gerard Kotte (NMi VSL) Netherlands
- \* Li He (NIM) China
- \* Samuel R. Low (NIST) USA
- \* Renato Reis Machado (INMETRO) Brazil Felix Meli (OFMET) Switzerland Leslie R.Pendrill (SP) Sweden Patrick Pinot (BNM-INM) France
- \* Alfredo Esparza Ramírez (CENAM) Mexico Robert Spurný (SMU) Slovakia
- \* Wayne Stiefel (NIST) USA
- *Mitsuru Tanaka* (NMIJ) Japan *Andrew Wallard* (BIPM) France

### Main topics of the meeting:

- 1. Key-comparisons: Vickers (doc: WGH-2004-Vickers draft B) and Brinell;
- 2. New definition for Rockwell hardness (doc: WGH-2004-02);
- 3. Procedure for testing Rockwell diamond indenters and machines (doc: WGH-2004-01);
- 4. Proposal for new Key-comparison on Rockwell C scale (doc: WGH-2004-Rockwell C TP);
- 5. Pilot study on Martens Hardness
- 6. Pilot study on Diamond Rockwell Indenters (doc: WGH-2004-Rockwell indenters TP);
- 7. Status of CMC's (MRA Appendix C)
- 8. Other business and next meeting

### Invited participants present at the meeting

Laurence Brice (NPL) United Kingdom Benny Burke (CSIR) South Africa Robert Ellis (D. L. Ellis) USA Li Ma (NIST) USA John Song (NIST) USA Anton Štibler (ZAG) Slovenia Satoshi Takagi (NMIJ) Japan

### 1. Key Comparisons

### Vickers (doc: WGH-2004-Vickers draft B);

Pilot laboratory PTB. Final Report, 2. Revised Draft B, has been completed. Was a unanimous decision to submit the report to Metrologia.

Recommendations at the end of the CCM Vickers key comparison:

- It was estimated that the tip radius of typical good indenters are 200 to 400 nm for micro-Vickers; 100 nm for Berkovich and 50 nm for nano-indenter. A tip is usually not spherical, but rather it resembles the irregular shape of a "potato."
- There were differing opinions expressed on the level of the influence of the tip radius on the hardness result.
- Tip radius is measured by scanning force microscope (SFM) at PTB, and by atomic force microscope (AFM) at NMIJ. Stylus measuring systems may not be capable of measuring the radius of indenter tips.
- WGH members are asked to send results of any investigations of Brinell, Rockwell and Vickers hardness influence parameters including the methods used (experimental or theory). The summarized results will be published on NPL website with a link from the BIPM website.

### Brinell

Status of the Brinell Key Comparison (see Appendix C).

- Pilot laboratories are KRISS (Korea) and NMIJ (Japan)
- Measurements are near completion.
- Hardness measurements are made on test blocks and diameter measurements are made on a glass plate having a thin film coating with holes of four sizes.

### 2. New definition for Rockwell hardness (doc: WGH-2004-02)

An important goal undertaken by the WGH is to reduce the differences in Rockwell hardness measurements made by National Metrology Institutes by better defining the Rockwell hardness test.

Following the discussion to define important parameters of the <u>Rockwell C scale</u> (HRC) test using document WGH-2004-02 as a reference (*see Appendix D*), the proposed reference values will be balloted by all WGH members and, if approved, will be submitted to the CCM-CIPM for approval.

	Proposed Refere	ence values for HRC		
	Test parameters	Reference value	Start measurement	Stop measurement
T.1	Preload application time $(T_{ap})$ $T_{preload} = \frac{T_{ap}}{2} + T_{rp}$	35	~1% of the force of preload	~99% of the force of preload
Т.2	Preload reading time (T <sub>rp</sub> ) $I_{preload} = \frac{1}{2} + I_{rp}$	35	~99% of the force of preload	Reading
Т.5	Full load dwell time (T <sub>dl</sub> )	5s	~99% of the force of full load	~99% of the force of full load
Т.6	Final reading time (T <sub>rf</sub> )	4s	~101% of the force of full load	~99% of the force of full load
V.4	Final mean_velocity of additional load application $(V_{fal})$	0,030mm/s	~80% of the force of preload	~99% of the force of full load
C.1	Temperature of test (T)	23°C	Beginning of the test	Beginning of the test
F.1	Preliminary force value (F <sub>0</sub> )	98,0665 N		
F.2	Total force value $(F_1)$	1470,998 N		
I.1	Average radius spherical tip (R <sub>a</sub> )	0,200 mm	-30° (from the axis)	$+30^{\circ}$ (from the axis)
I.2	Average cone angle $(\alpha_m)$	120°	±30°	±400 µm

### 3. Procedure for testing Rockwell diamond indenters and machines (doc: WGH-2004-PS Rock Dia Ind.doc)

Hysteresis tests on indenters indicating that hysteresis is different among indenters have been presented and discussed.

It has been proposed a Pilot Study (*see Appendix E*) amongst the NMIs to compare the methods being used to measure indenter geometry, and to compare the results of performance studies of indenter hysterysis.

• The Pilot Study will involve the circulation of two/three HRC blocks (different hardness levels) and two common indenters among the NMIs:

• The following NMIs agreed to participate	
CENAM, Mexico	
CSIR, South Africa (tentative)	NIST, USA
INMETRO, Brazil	NMIJ, Japan
IMGC, Italy	NPL, UK
KRISS, Korea	PTB, Germany
NIM, PR China (tentative)	VNIIFTRI, Russia

### 4. Proposal for new Key-comparison on Rockwell C scale (doc: WGH-2004-Rockwell C TP);

In light of the new definition proposed to be adopted by NMIs (*see section 4 above*), a comparison of the Rockwell C Hardness scale realized by primary hardness Rockwell machines is necessary to verify the implementation of the new definition.

- It is proposed (*see Appendix F*) to compare five hardness level equally distributed along the Rockwell C scale.
- In the comparison one common indenter is used to separate the indenter effect, as well the institute indenter for each participant.

The pilot laboratories will be IMGC/NIST/NMIJ/PTB. The pilot laboratories will supply hardness blocks and the common indenter for the comparison.

The participation to the comparison is opened to all NMIs that satisfy rules for the participation to the Key comparison written in MRA and in the Appendix of the "*Guidelines for CIPM key comparison*" March 1999 (see annex).

### 5. Pilot study on Martens Hardness

It was decided to have a Martens hardness Pilot Study at the Torino, Italy meeting (2001). Following that meeting, a survey concerning the Pilot Study was circulated. The conclusion was that members are more interested in nano-hardness. Hence, the Martens hardness Pilot Study is cancelled and will be substituted with a nano-hardness Pilot Study. The nano-hardness Pilot Study is currently being prepared by KRISS (Korea)

### 6. Pilot study on Diamond Rockwell Indenters

The Pilot Study was discussed in Section 3 above.

### 7. Status of CMC's (MRA Appendix C)

NMIs are asked to contact their Regional Metrology Organizations to revise their CMC tables in light of the results of a new key comparison.

There was a discussion of how to report hardness uncertainty. The issue in question is whether to include an uncertainty component due to block non-uniformity or simply state the uncertainty of the machine, operator, environment, etc. Also, if it is to be included, on what should it be based? For example, should it be based on a perfectly uniform block (zero uncertainty), or the best block ever measured by the laboratory, or the best block known to exist?

Another issue that was discussed is - when reporting a uncertainty for a range of a hardness scale, what uncertainty should be reported; the lowest, the highest or an uncertainty range that can be correlated to a specific hardness value?

All NMIs are asked to consult with their Regional Metrology Organization concerning the above questions and report this information to the chairman All NMIs are also asked to report to the chairman the best (lowest) block uncertainty that they have measured.

### 8. Other business and next meeting

results of a bilateral comparison between VNIIFTRI (Russia) and PTB (Germany) for the HV 1 and VV 30 hardness scales (*see Appendix G*) have been presented. The conclusion of the study was that the comparison is considered successful and will provide a link to the key comparison.

The next meeting of the WGH will be on the  $12^{th}$  or  $13^{th}$  October 2005 at NPL, Teddington, UK during the meetings week of ISO TC 164/ SC 3.

**Appendix B:** Recommendations (Section 10) given in CCM Vickers key comparison, Final Report, 2. Revised Draft B

### 10. Discussions, conclusions and remarks

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 load 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  $d > 100 \mu m$  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 about the relationships between both methods.

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

Appendix C: Information from K. Hattori's presentation of the status of the current Brinell Key Comparison

**Brinell Key Comparison KRISS/NMIJ** K. Hattori, NMIJ

### Introduction

- The Brinell KC started at Nov. 2003. •
  - Pilot laboratory KRISS and NMIJ
- Items used in this comparison •
  - Hardness and levels
    - o 250HBW1/30, 250HBW2.5/187.5, 250HBW5/750
    - o 350HBW1/30, 350HBW2.5/187.5, 350HBW5/750, 350HBW10/3000
    - o 450HBW1/30, 450HBW2.5/187.5, 450HBW5/750, 450HBW10/3000
  - False indentation
    - Holes on thin metal films on glass plates
      - Provided by IMGC. thank you Dr. Germak! •
    - o 0.24, 0.4, 0.8, 1.5mm in diameter.

### Status

Participant	Received	Send	Data report
NMIJ	-	27 Nov. 2003	27 Nov. 2003
NIM	04 Dec.2003	08 Jan. 2004	in progress
VNIIFTRI	06 Feb. 2004	24 Feb. 2004	03 Mar. 2004
IMGC	15 Mar. 2004	07 Apr. 2004	5-May-04
PTB	xx xxx. 2004	18. May. 2004	19-May-04
NPL	20-May-04	15-Jul-04	26 Aug. 2004
KRISS	02 Aug. 2004	02 Aug. 2004 04 Oct. 2004 in pi	
NMIJ	19 Oct. 2004	-	Meas. in progress

## CIPM Rockwell C hardness scale definition

One of the limits of hardness quantity is its conventional definition. At the present, both calibration laboratories and primary laboratories use the same definition (ISO 6508-part3) in terms of value of the parameters of the hardness test and their related tolerances. Same definition gives the same level of uncertainty.

Present definition (ISO) has some lacks in the identification of the influence parameters of the hardness test and some of them are not metrologically well defined.

An analysis of the present definition has been done and presented to the 5<sup>th</sup> CCM-WGH meeting in Rio de Janeiro in May 2002.

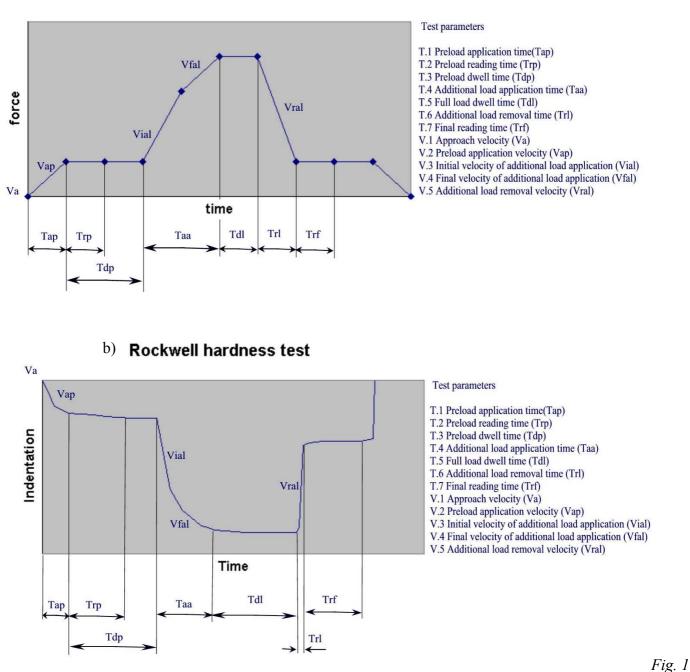
A questionnaire was circulate to investigate the possibility of the primary hardness standard machines to control and measure the influence parameters (annex A).

Selecting few, but the most important influence parameters to be controlled (fig. 1), following, a proposal for a CIPM definition for the Rockwell C scale to be used by the NMIs is presented:

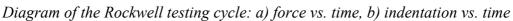
	Test parameters	Reference value	Start measurement	Stop measurement
T.1	Preload application time $(T_{ap})$ $T$ $T_{ap}$ $T$	2	~1% of the force of preload	~99% of the force of preload
Т.2	$\frac{(T_{ap})}{Preload reading time} T_{preload} = \frac{T_{ap}}{2} + T_{rp}$ $(T_{rp})$	3s	~99% of the force of preload	Reading
Т.5	Full load dwell time (T <sub>dl</sub> )	5s	~99% of the force of full load	~99% of the force of full load
Т.6	Final reading time (T <sub>rf</sub> )	4s	~101% of the force of full load	~99% of the force of full load
V.4	Final mean_velocity of additional load application ( $V_{fal}$ )	0,030mm/s	~80% of the force of preload	~99% of the force of full load
C.1	Temperature of test (T)	23°C (or 20°C)	Beginning of the test	Beginning of the test
F.1	Preliminary force value (F <sub>0</sub> )	98,0665 N		
F.2	Total force value (F <sub>1</sub> )	1470,998 N		
I.1	Average radius spherical tip (R <sub>a</sub> )	0,200 mm	-30° (from the axis)	+30° (from the axis)
I.2	Average cone angle $(\alpha_m)$	120°	±30°	±400 µm

This kind of definition allow every laboratory to calculate the uncertainty following the ISO GUM specifications.

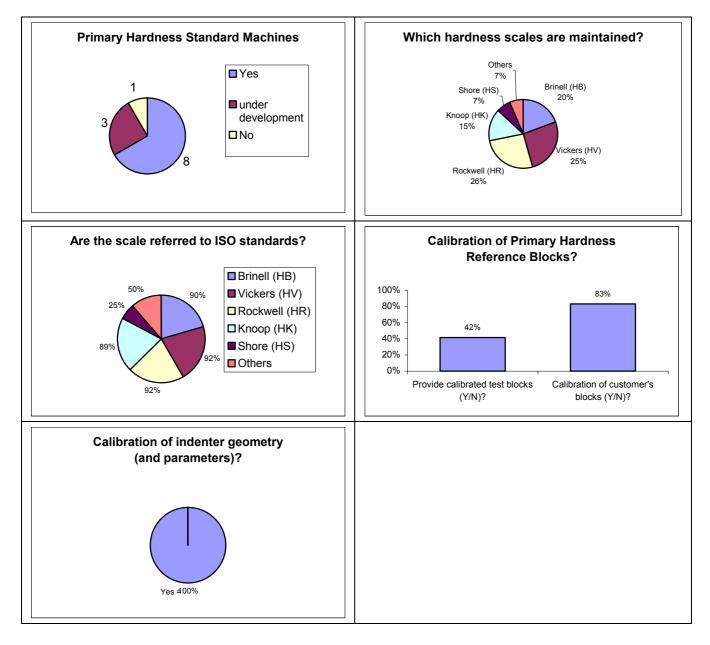
An example of uncertainty calculation is given in annex B as MS-EXCEL® file where, for any influence parameter, an estimation of the contribution to the uncertainty is calculated. A table with the recommended sensitivity coefficients is also given in Annex B.



### a) Rockwell hardness test



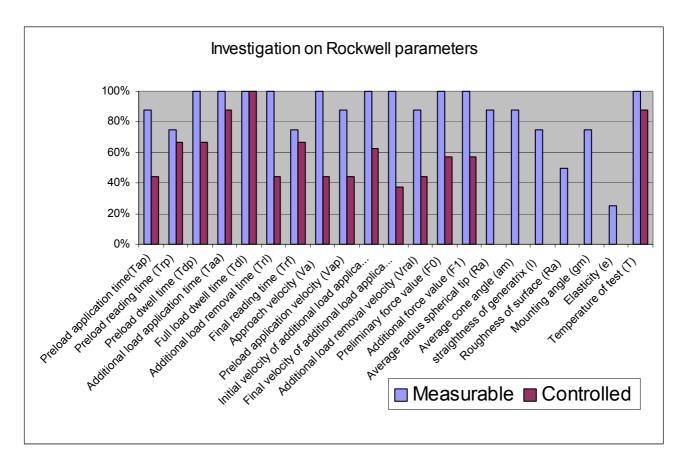
### ANNEX A Analysis of the WGH Questionnaire submitted to the NMIs (2002)

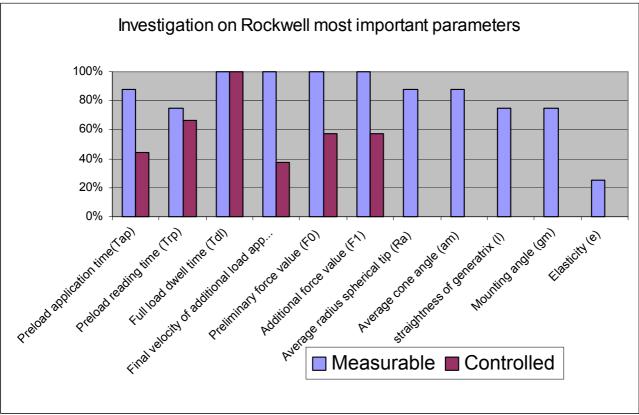


		Measurable	Controlled	Range	Used reference	ISO
		Y/N	Y/N	Min/Max	values	Specifications
For	ce parameters					
T.1	Preload application time(T <sub>ap</sub> )	Y=7 N=1	<mark>Y=4 N=5</mark>	<mark>0s / 60s</mark> 0,1s / ∞ (2) 2s / 6s	None reported	
<mark>T.2</mark>	Preload reading time $(T_{rp})$	Y=6 N=1	<mark>Y=6 N=2</mark>	0s / 60s 0,1s / ∞ (2) 1s / 70s 1s / 99s	2s 3s (3) 5s (2)	<mark>&lt;3 s</mark>
<mark>T.3</mark>	Preload dwell time (T <sub>dp</sub> )	<mark>Y=8 N=0</mark>	<mark>Y=6 N=3</mark>	0s / 20s 2s / ∞ (2) 2s / 3s 1s / 70s ? / 1s	2 s (2) 5 s (3)	
T.4	Additional load application time (T <sub>aa</sub> )	Y=8 N=0	Y=7 N=1	0s / 60s 1s / ∞ (2) 1s / 30s ? / 6s	1 s to 8 s (3) 3 s 6 s 10 s	(1÷8) s
<mark>T.5</mark>	Full load dwell time (T <sub>dl</sub> )	Y=8 N=0	Y=9 N=0	0s / 60s 1s / ∞ (2) 1s / 99s 1s / 70s 1s / 99s	2 s to 6 s 3 s 4 s 5 s (2) 5,5 s 6 s 10 s	<mark>4±2 s</mark>
T.6	Additional load removal time (T <sub>rl</sub> )	Y=8 N=0	Y=4 N=5	$\begin{array}{c} 0,5s \ / \ 2s \\ 0s \ / \ 60s \\ 1s \ / \ \infty \ (2) \\ 4s \ / \ 5s \\ ? \ / \ 6s \end{array}$	5s 4s	
T.7	Final reading time $(T_{rf})$	Y=6 N=1	Y=6 N=2	$\begin{array}{c} 0s \ / \ 10s \\ 0,1s \ / \ \infty \ (2) \\ 1s \ / \ 70s \\ 1s \ / \ 99s \end{array}$	1 s 3 s 4 s 5 s (4)	(3÷5) s
Ve	locity parameters					
	Approach velocity (V <sub>a</sub> )	Y=8 N=0	Y=4 N=5	0 mm/s to 5 mm/s 1 µm/s to 1 mm/s 1 µm/s to 5 mm/s ? to 0,8 mm/s 1 µm/s to 1 mm/s	< 1 mm/s 100	<1 mm/s
V.2	Preload application velocity $(V_{ap})$	Y=7 N=1	Y=4 N=5	1 μm/s to 1 mm/s 1 μm/s to 1 mm/s	0,5 mm/s	
V.3	Initial velocity of additional load application (V <sub>ial</sub> )	Y=8 N=0	Y=5 N=3	1 μm/s to 1 mm/s 5 μm/s to 120 μm/s 1 μm/s to 1 mm/s	(50 to 100) μm/s 40 μm/s ~500 μm/s	
	Final velocity of additional load application (V <sub>fal</sub> )	<mark>Y=8 N=0</mark>	<mark>Y=3 N=5</mark>	1 μm/s to 1 mm/s 5 μm/s to 120 μm/s 1 μm/s to 1 mm/s	(20 to 40) μm/s 20 μm/s (50 to 100) μm/s 40 μm/s	<mark>(20 to 40) µm/s</mark>
V.5	Additional load removal velocity (V <sub>ral</sub> )	Y=7 N=1	Y=4 N=5	1 μm/s to 1 mm/s 1 μm/s to 1 mm/s	~500 µm/s	

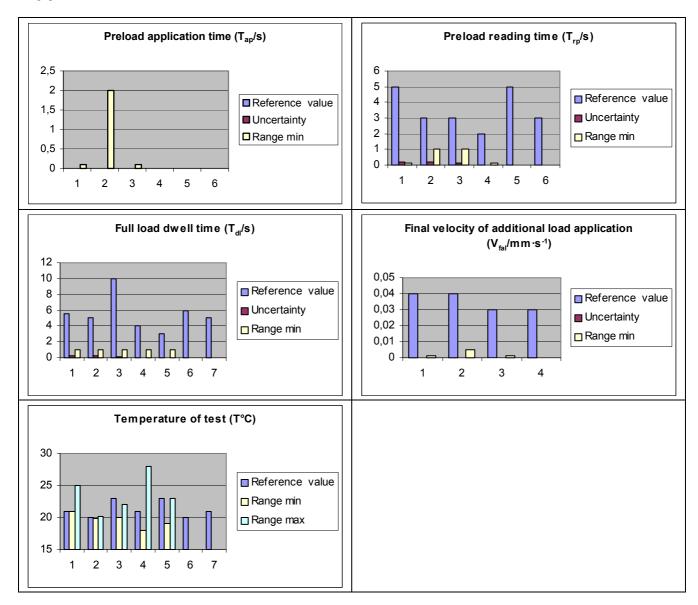
### Appendix D: WGH-2004-HRC def.doc9 Nov. 2004 Rev. 01

		Measurable	Controlled	Range	Used reference	ISO
		Y/N	Y/N	Min/Max	value	Specifications
For	ce parameters					
F.1	Preliminary force value (F <sub>0</sub> )	<mark>Y=8 N=0</mark>	<mark>Y=4 N=3</mark>	0,1 N to 200 N 29,42 N to 98,07 N 0,1 N to 200 N	98,07 N ± 0,2 N	98,07 N ± 0,2 N
F.2	Additional force value (F <sub>1</sub> )	<mark>Y=8 N=1</mark>	<mark>Y=4 N=3</mark>	0 N to 2000 N 117,7 N to 1373 N 0 N to 2000 N	1373 N ± 1,4 N	1373 N ± 1,4 N
Inc	lenter parameters					
<mark>l.1</mark>	Average radius spherical tip (R <sub>a</sub> )	<mark>Y=7 N=1</mark>	<mark>Y=0 N=5</mark>	30 to 100 mm	$\begin{array}{l} 0,2 \ \text{mm} \pm 0,002 \ \text{mm} \\ 0,2 \ \text{mm} \pm 0,005 \ \text{mm} \\ 0,2 \ \text{mm} \pm 0,01 \ \text{mm} \end{array}$	<mark>0,2 mm ± 0,005 mm</mark>
<mark>I.2</mark>	Average cone angle $(\alpha_m)$	<mark>Y=7 N=1</mark>	<mark>Y=0 N=5</mark>	<mark>1° to 180°</mark>	$\frac{120^{\circ} \pm 0,05^{\circ}}{120^{\circ} \pm 0,1^{\circ}}$ $\frac{120^{\circ} \pm 0,1^{\circ}}{120^{\circ} \pm 0,1^{\circ}}$	$120^{\circ} \pm 0,1^{\circ}$
1.3	straightness of generatrix $(\lambda)$	Y=6 N=1	Y=0 N=5	0.1 to 60	0,25 μm per 0,4 mm 0,0005 mm	
1.4	Roughness of surface (Ra)	Y=4 N=4	Y=0 N=5	0.01 to 1	Rm < 0,05 mm	
1.5	Mounting angle $(\gamma_m)$	Y=6 N=2	Y=0 N=5	0,05° to 2°	<= 0,8° 0,3° < 25'	
I.6	Elasticity (ε)	Y=2 N=6	Y=0 N=5			
Te	mperature parameter	'S				
C.1	Temperature of test (T)	Y=8 N=1	<mark>Y=7 N=1</mark>	19 / 23 18 / 28 20 / 22 19.8 / 20.2 19 / 23	21°C ± 1,5°C 20°C 21°C 23°C ± 5°C (2) 20°C 21°C ± 2°C	<mark>23°C ± 5°C</mark>





### Appendix D: WGH-2004-HRC def.doc9 Nov. 2004 Rev. 01



### ANNEX B Evaluation of the uncertainty - Example

	C	ertificate da	ta		Mea	asured hardr	less
Xi	$\Delta x_i$	<i>U</i> i (2 <i>□</i> )	□i	$c_i = \frac{\Delta H}{\Delta x_i}$	$\Delta H_i$	u;²(H)	u <sub>i</sub> ⁴( <b>H)/</b> □ <sub>i</sub>
				,	HRC	HRC <sup>2</sup>	$HRC^{4}$
F <sub>0</sub> /N	0.01	0.01	20	1.2E-01	1.2E-03	3.6E-07	6.5E-15
F/N	0.15	0.05	20	-4.0E-02	-6.0E-03	1.0E-06	5.0E-14
<i>□</i> /°	0.05	0.02	20	1.3E+00	6.5E-02	1.7E-04	1.4E-09
<i>r/</i> mm	0.003	0.001	20	1.5E+01	4.5E-02	5.6E-05	1.6E-10
<i>h/</i> (µm)	0.1	0.05	20	-5.0E-01	-5.0E-02	1.6E-04	1.2E-09
<i>v/</i> (µm/s)	5	2	10	-2.0E-02	-1.0E-01	4.0E-04	1.6E-08
t <sub>o</sub> /s	0.5	0.2	10	1.0E-02	5.0E-03	1.0E-06	1.0E-13
t/s	0.5	0.2	10	-7.0E-02	-3.5E-02	4.9E-05	2.4E-10
Total					-0.07	0.001	1.9E-08
Standard u	ncertainty	$u_m/HRC$				0.03	
Coverage f	actor k for a		2.03				
Expanded uncertainty $U/\text{HRC} = ku/\text{HRC}$ 0.06							
Where 2	$\Delta H_i = c$	$_{i}\Delta \mathbf{x}_{i}$ and	d $u_i^2$	$^{2}(H)\approx c_{i}^{2}u$	$^{2}(X_{i})$		

# Example of evaluation of the uncertainty of primary hardness standard machines for the 20 HRC to 25 HRC hardness level.

Recommended Sensitivity Coefficients for Rockwell C scale:

Sensitivity Coefficients $c_i = \frac{\Delta H}{\Delta x_i}$								
$X_i$		Hardness leve	1					
Λi	(20-25)/HRC	(40-45)/HRC	(60-65)/HRC					
<i>F<sub>0</sub></i> /N	1,2E-01	7,0E-02	5,0E-02					
<i>F</i> /N	-4,0E-02	-3,0E-02	-2,0E-02					
αl°	1,3E+00	8,0E-01	4,0E-01					
<i>r</i> /mm	1,5E+01	3,0E+01	5,0E+01					
<i>h</i> /(µm)	-5,0E-01	-5,0E-01	-5,0E-01					
<i>v</i> /(µm/s)	-2,0E-02	0,0E+00	3,0E-02					
t₀/s	1,0E-02	5,0E-03	4,0E-03					
<i>t</i> /s	-7,0E-02	-4,0E-02	-3,0E-02					

Appendix E: WGH-2004-PS Rock Dia Ind.doc 8 Nov. 2004 Rev. 00

### Pilot study on Rockwell diamond indenters

Much work has been done on Rockwell diamond indenters to evaluate the relationship between geometry and hardness measurement results. However, it has been observed that the residual after geometrical correction is significant (within  $\pm 0.3$  HRC).

The reason for these residuals is not completely clear. At the present level of knowledge, it seems that it is not possible to predict the performance of a diamond indenter using <u>only direct measurement of the geometry</u>.

In fact, ISO Standards prescribe that indenters, used in calibrations of hardness blocks, must be verified by comparison with so called "<u>reference indenters</u>" recognized at the national level ("<u>metrological indenters</u>"). Metrological indenters <u>are not defined</u> at all by the ISO standards or by any other international organization.

To select suitable metrological indenters, it is necessary to perform <u>both geometrical</u> <u>measurements and performance tests</u>.

- <u>Geometrical measurement</u> can be done in different ways, but to assure result compatibility, it is necessary to compare the definition of the measurand and the measurement capabilities. The compatibility assurance will probably be obtained by the results of comparisons among some NMIs.
- <u>Performance tests</u> require the use of hardness standardizing machines working with an agreed measurement cycle and knowing the machine's performance with respect to internationally agreed hardness scales.

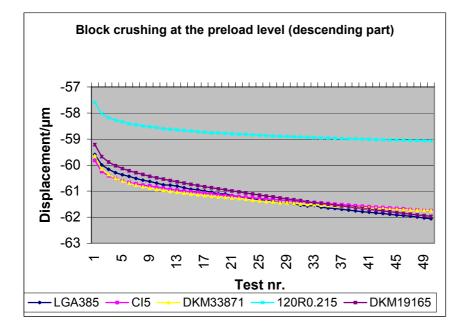
It is therefore important to develop a procedure for qualifying metrological indenters, so that any NMI maintaining hardness scales could procure his primary indenters.

An effort has been made to acquire a selection of National "Metrological" Rockwell diamond indenters. In order to accomplish this goal, both geometrical characterization and performance comparisons of the indenters have been performed.

IMGC has made hysteresis tests on indenters indicating that hysteresis is different among indenters.

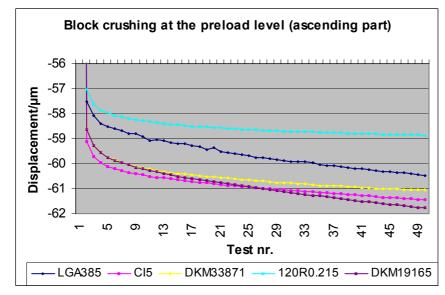
Description of the test:

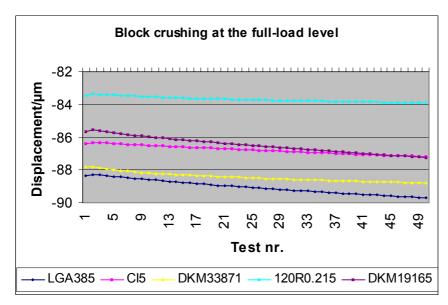
- 50 Rockwell C tests on the same indentation (different hardness level), recording the indentation levels at the preload (descending and ascending part) and at the full-load
- Calculation of the indenter hysteresis as differences from the displacement measured during the descending and ascending preload.



#### Fig. 1

Performance test of the indenters: recording of the block crushing at the preload level (descending part) during 50 Rockwell C tests (about 70HRC hardness level) on the same indentation made with five different indenters.



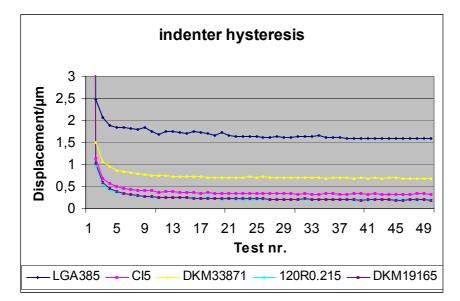


#### Fig. 2

Performance test of the indenters: recording of the block crushing at the preload level (ascending part) during 50 Rockwell C tests (about 70HRC hardness level) on the same indentation made with five different indenters.

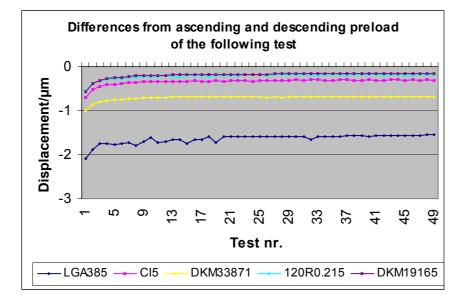
### Fig. 3

Performance test of the indenters: recording of the block crushing at the full-load level during 50 Rockwell C tests (about 70HRC hardness level) on the same indentation made with five different indenters.



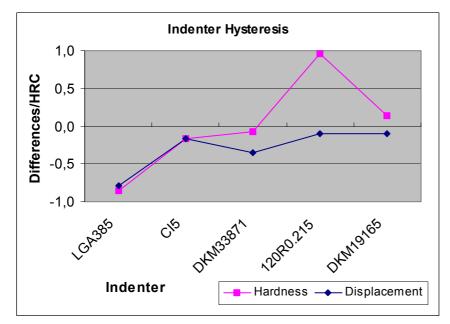
#### Fig. 4

Performance test of the indenters: evaluation of the indenter hysteresis calculated as difference from the displacement measured during the descending and ascending preload.



#### Fig. 5

Performance test of the indenters: difference from the displacement measured during the ascending and descending preload of the following test.



### Fig. 6

Performance test of the indenters: comparison between the differences measured in Hardness (Hardness) and those calculated as hysteresis and converted in Hardness (Displacement).

Note that the hardness measurement results are not corrected for the geometry of the indenter (indenter 120R0.215 means an indenter with 120° angle and 215µm radius).

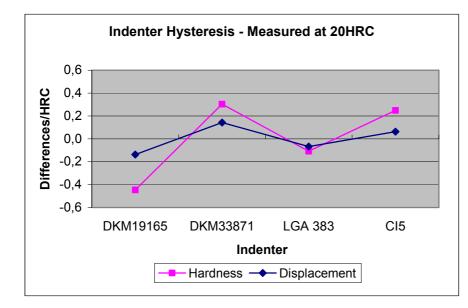
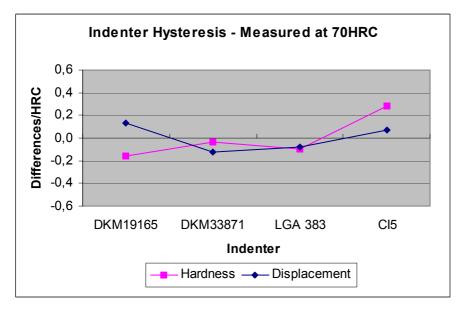


Fig. 7

Performance test of the indenters: hysteresis calculated (and converted in hardness – Displacement) using and 20HRC block and comparison the differences measured on the hardness measurements (Hardness).

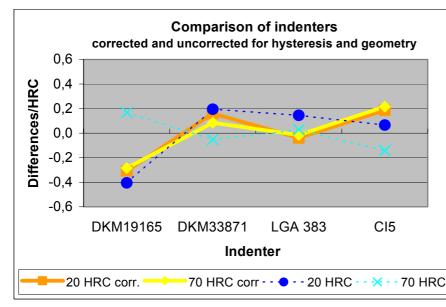
Hardness measurements are corrected for the geometry of the indenters.



#### Fig. 8

Performance test of the indenters: hysteresis calculated (and converted in hardness – Displacement) using and 70HRC block and comparison the differences measured on the hardness measurements (Hardness).

Hardness measurements are corrected for the geometry of the indenters.



#### Fig. 9

Performance test of the indenters: results of the comparison of indenters corrected for both hysteresis and geometry at 20HRC and 70HRC levels.

### Proposal:

a circulation of two/three HRC blocks (different hardness level) and two common indenters among the NMIs.

All Institutes measure the geometry of the indenter/s and carry out the performance test as described previously on two common indenters and own national indenter with their machine.

A comparison of results of geometrical measurements and of performance tests are made to assure:

- 1) Compatible measurements of geometrical characteristic of the indenters (inside the declared uncertainty)
- 2) Application of the performance test on all type of machines

### **Final discussion:**

- possibility to apply a correction (geometry-hysteresis) for metrological indenters.

## Rockwell C Hardness scale Key-Comparison

### - Proposal -

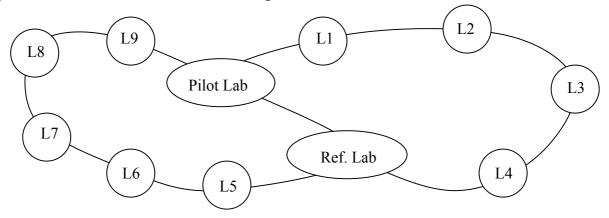
At the light of the new definition proposed to be adopted by NMIs (see "*WGH-2004-HRC def*" document), a comparison of the Rockwell C Hardness scale realized by primary hardness Rockwell machines is necessary to verify the implementation of the new definition.

- It is proposed to compare five hardness level equally distributed along the Rockwell C scale.
- In the comparison one common indenter is used to separate the indenter effect, as well the institute indenter for each participant.

The pilot laboratories will be IMGC/NIST/NMIJ/PTB. The pilot laboratories will supply hardness blocks and the common indenter for the comparison.

The participation to the comparison is opened to all NMIs that satisfy rules for the participation to the Key comparison written in MRA and in the Appendix of the "*Guidelines for CIPM key comparison*" March 1999 (see annex).

In the case the set of five blocks is not sufficient to perform all measurements for the whole comparison, different set of blocks will circulate among sub-set of laboratory; Pilot Laboratory, and eventually another laboratory (Reference lab.), will be the conjunction point to allow the link between the subcomparisons, as shown in the following scheme:



### Annex

### Note on eligibility for participation in

### a CIPM or RMO key comparison

The following is an extract from the agreement on mutual recognition of national measurement standards and calibration certificates issued by national metrology institutes:

### 6. Participation in key and supplementary comparisons

6.1 Participation in a CIPM key comparison is open to laboratories having the highest technical competence and experience, normally the member laboratories of the appropriate Consultative Committee. Those laboratories that are not members of a Consultative Committee and not NMIs must be nominated by the designated national metrology institute referred to in pararagraph 1.4. as being responsible for the relevant national measurement standards. In choosing participants, the Consultative Committees must take proper account of regional representation. The number of laboratories participating in CIPM key comparisons may be restricted for technical reasons.

6.2 Participation in key comparisons organized by an RMO is open to all RMO members and to other institutes that meet the rules of the regional organization (including institutes invited from outside the region) and that have technical competence appropriate to the particular comparison.

6.3 Participation in RMO supplementary comparisons is open to those institutes meeting the requirements specified in paragraph 6.2.

COOMET.M.H-K1.b и COOMET.M.H-K1.c Vickers PTB / VNIIFTRI comparison

Participants:

- Physikalisch-Technische Bundesanstalt, PTB
- All-Russian Scientific Research Institute for Physical-Technical and Radiotechnical Measurements, VNIIFTRI

Pilot laboratory - PTB, Braunschweig

Three sets of hardness reference blocks for the Vickers hardness scales HV1 and HV30 consisting each of three hardness reference blocks with the hardness levels 240 HV, 540 HV and 840 HV are used.

- The dimensions are length 60 mm, width 60 mm, thickness 10 mm.
- The blocks are manufactured as commercial products by Buderus Co., Germany.
- A grid with  $13 \times 13 = 169$  fields was engraved on the measurement surface of each block.

The measurands used in this comparison were of two kinds:

- 1. the mean value each of eight hardness measurements on a hardness reference block
- 2. the mean diagonal length of six reference indents had to be determined according to ISO 6507-1 and -3.

The hardness measurements and reference indents were made in the hardness scales HV1 and HV30 each for the nominal hardness levels 240 HV, 540 HV and 840 HV.

Table 1 – Mean values comparison (in HV)										
Hardness scale	Value PTB	Uncert. PTB	Value VNIIFTRI	Uncert. VNIIFTRI	Difference VNIIFTRI - PTB	Acceptable difference				
240 HV1	244,06	7,5	238,80	8,3	-5,26	5,97				
240 HV30	238,00	2,1	238,52	2,4	0,52	1,85				
540 HV1	539,03	17,9	534,50	23,9	-4,53	13,87				
540 HV30	522,89	6,9	524,38	7,0	1,49	6,33				
840 HV1	831,49	30,1	830,63	38,1	-0,86	22,49				
840 HV30	817,86	9,9	818,13	14,0	0,27	11,79				

### RESULTS

### Table 2 - Evaluation of the reference indentation(in µm)

Ref. Indents	Value PTB	St. Dev. PTB	Value VNIIFTRI	Std. Dev. VNIIFTRI	Difference VNIIFTRI -PTB	Acceptable difference
240 HV1	87,55	0,05	88,40	0,30	0,85	0,61
240 HV30	482,48	0,68	482,40	1,07	-0,08	2,54
540 HV1	58,58	0,12	58,35	0,16	-0,23	0,40
540 HV30	326,03	0,82	323,50	1,32	-2,53	3,11
840 HV1	47,38	0,11	47,15	0,19	-0,23	0,44
840 HV30	259,35	0,22	259,50	1,01	0,15	2,07

**Appendix G:** Information from E. Aslanyan's presentation of Vickers PTB/VNIIFTRI bilateral comparison

# Results of the comparison between VNIIFTRI and reference values of CCM Vickers key comparison

Evaluation of the hardness values				(Values in HV)		
Hardness scale	Ref. Value	Unc. Ref. Val.	Value VNIIFTRI	Unc.VNIIFTRI	Diff.(VNIIFTRI-Ref.Val.)	Acceptable diff.
240 HV1	240.09	1.17	238.80	3.32	-1.29	3.52
240 HV30	237.73	0.60	238.52	1.18	0.79	1.32
540 HV1	534.77	3.52	534.50	8.48	-0.27	9.18
540 HV30	522.95	1.85	524.38	4.40	1.43	4.77
840 HV1	828.49	6.63	830.63	13.18	2.14	14.75
840 HV30	815.42	3.52	818.13	7.82	2.71	8.58

CONCLUSION

- The COOMET Vickers PTB/VNIIFTRI comparison can be considered as a successful metrological exercise.
- The contribution of this comparison would be quite important because other COOMET countries need the confirmation of traceability by a key comparison.