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EXPERIENCE IN THE METROLOGICAL CHARACTERIZATION OF PRIMARY HARDNESS STANDARD MACHINES

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Abstract – The Istituto Nazionale di Ricerca Metrologica (INRIM) and Galileo section of LTF S.p.a. have cooperated for many years in the field of hardness for developing and improving Primary Hardness Standards and measuring systems for their laboratories. With this experience, Galileo-LTF has realized many installations for several NMIs in the world. All these Hardness Standards and measuring systems have been metrologically characterised by INRIM. In the paper, experiences made during the metrological characterization will be shown. They include the methods and results of direct verification of influence parameters (force, displacement, time, velocity, angle, etc.) and of indirect verification (hardness scales or indenters comparison) of the systems.

Keywords: hardness, primary standards, measuring systems

1. INTRODUCTION

The activity of realization and improvement of Primary Hardness Standard Machines (PHSM) and related measuring systems began, at Istituto Nazionale di Ricerca Metrologica (INRIM) (formerly Istituto di Metrologia "G. Colonnetti" - IMGC) in Torino (Italy), in the ’70 years of the past Century. With the cooperation of Galileo section of LTF S.p.a. in Antegnate (Italy), which acquired patents and know-how, this activity continued until the present days [1].

The most important result of this cooperation has been, from one side, the possibility for developing and improving the INRIM hardness standards and measuring systems, as well the LTF hardness standards and measuring systems for it accredited calibration laboratory, and, on the other side, the possibility to develop new standards and measuring systems for other National Metrology Institutes (NMIs).

Starting from the first realizations for NMIs, like the PHSM for NIST in USA [2] and the automatic measuring system and the indenter measuring system for NPL in UK [3], in the last two years several primary standards and measuring systems have been supplied to many NMIs in the world. In detail:

- three PHSMs for INMETRO (Brazil), NCM (Bulgaria), NPL (India),
- one Vickers hardness calibration machine for NIM (China),
- five automatic measuring systems for Vickers and Brinell indentations for INEMTRO (Brazil), NCM (Bulgaria), NPL (India), UME (Turkey), NIM (China),
- two measuring systems for the geometric characterization of Rockwell and Vickers indenters for INMETRO (Brazil), UME (Turkey).

All these standards and measuring systems have been realized by LTF and metrologically characterised by INRIM [4-10].

The reported expanded uncertainties of measurement U are calculated following the relative standards and international documents [11-12] and they are stated as the standard uncertainty of measurement multiplied by the appropriated coverage factor corresponding to a coverage probability of 95%.

The results have been compared with technical specifications of measuring systems.

2. PRIMARY ROCKWELL HARDNESS STANDARD MACHINES (PHSMs)

The INRIM PHSM was design and realized the end of the ’70 years of the last century [13-14]. It has been improved in electronic, electro-mechanics and software control in occasion of the second realization, at the beginning of ’90 years, for NIST (USA) [2]. New improvement was done during the third realization for the LFT accredited calibration laboratory at the beginning of the new century. In the last two years, three others PHSMs were realized for INMETRO (Brazil) (fig. 1), NCM (Bulgaria), NPL (India).

It consists in a dead weight machine for the generation of the test forces and a laser interferometric system for the indentation depth measurements. The main characteristics are also the high stiffness, isostatic design and a very flexible software control that permits to set and measure all the most important parameters involved in the test cycle (times and velocities)

The metrological characterization consists in the direct and indirect verification of the machine; in the direct verification, the main verified parameters are the forces generated by the PHSM and the geometry of indenters. The indirect verification consists in the comparison of the performances of indenters with the so called “National Indenters” and in the comparison of the hardness scales generated by the PHSM with the national scales maintained by INRIM.

2.1. Direct verification

2.1.1. Force

The INMETRO PHSM generate forces by means of dead weighs in the range from about 30N up to 1850N, the NCM PHSM in the range from about 30N up to 600N, the NPLI PHSM in the range from about 10 N up to 1 kN.

Fig. 1. INMETRO PHSM in the place of verification (INMETRO hardness laboratory - Brazil).

Two different type of frames have been used: in one case the weight of the frame is used to generate the force of preload for superficial Rockwell scales (about 30N) meanwhile in other case the weight of the frame is used to generate the force of preload for all other Rockwell scales (about 100N). In the first case the capacity of the PHSM is limited to 600N or 1kN, in the second to 1850N. In the second case, to reduce the force of preload for superficial Rockwell scales, three pulleys rotating on air bearing are used to balance the preload force up to about 30N.

PHSMs generate forces that, depending by the model, are used for the realization of all Rockwell scales, Vickers scales (from HV1 to HV100) and Brinell scales (from HB1/1 to HB2,5/187,5).

Two different load cells and one balance have been used for the direct verification of the forces generated by the PHSMs (figs. 2 and 3). Load cells and the balance with an adequate uncertainty have been calibrated at INRIM before the verification.

To perform the verification, the anvil of PHSMs has been removed to create room to lodge the load cells and the balance.

To guarantee that the PHSMs work correctly, the forces generated by the dead weights have been checked in different positions of the machine frame: +18 mm, +12 mm and +7 mm, correspondent at the whole interval which the machine works during the calibration activity due to the possible thickness differences of hardness blocks. This check is very important for all forces generated for the preload of the superficial scales, were the effect of the pulley to reduce the force is more significant.

Fig. 2. Balance used for the verification of the test forces for low loads (max 30N)

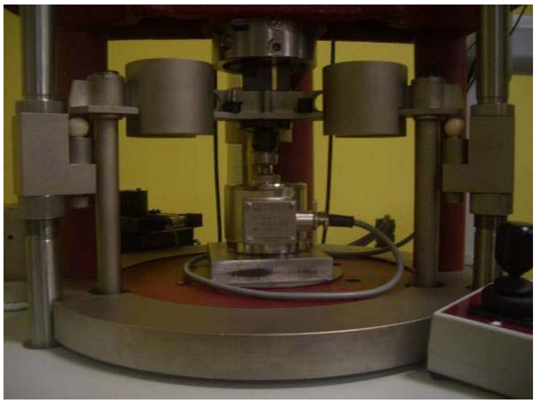

Fig. 3. Load cell used for the verification of the test forces

When the force measurements have been carried out at LTF laboratory, they have been corrected taking into the account the differences of the acceleration due to gravity, *g*, among INRIM, LTF in Italy, and the other NMIs Laboratories (Bulgaria, India or Brazil).

In fig 4 examples of results of the force verification are presented.

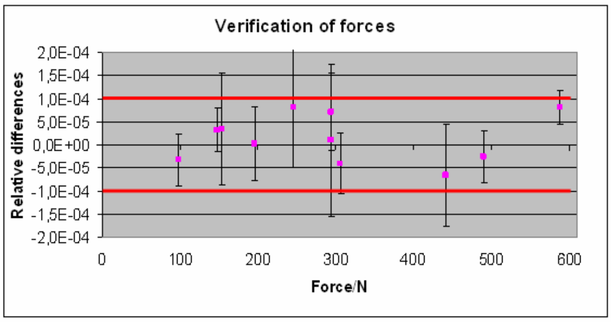

Fig. 4. Example of force accuracy of a PHSM.

As it is possible see in the figure, all measurement results are within the accuracy declared of the machine (100 ppm equal to $1x10^{-4}$). So, specifications of related standards [15-17] are largely satisfied.

2.1.2. Depth measuring system

The laser systems used for measuring the indentation depth have a calibration certificate declaring an uncertainty of $2x10^{-5}$. To assure the correct displacement measurement, the verticality of the laser beam and the perfect alignment of the interferometer have been verified.

To assure the verticality of the laser beam, a plane mirror orthogonal to the verticality as been create using an alcohol bath. The inclination of the interferometer has been adjusted until the coincidence between the source and reflected beam has been achieved. The estimation of the alignment in this kind of operation is 1mm on the coincidence between the laser beams on 260 mm of distance between the alcohol bath and the interferometer: it corresponds to an angle of 0,00385 rad. In these conditions, the possible relative cosine error in the indention depth is $7{,}4x10^{-6}$.

Finally, they have been verified by comparison with gage blocks lodged between the anvil and a false indenter (ceramic ball of 19 mm or 10 mm diameter) (fig. 5). The results, on a distance of about 9 mm (that represent the maximum range of the hardness block thickness) show a relative difference of some part in 10^6. It is negligible compared to the ISO6803-3 requirements ($1x10^{-3}$) and it corresponds, when the machine is measuring a depth of 180µm (maximum for Rockwell test), to about 0,01µm (within the resolution of the laser measuring system).

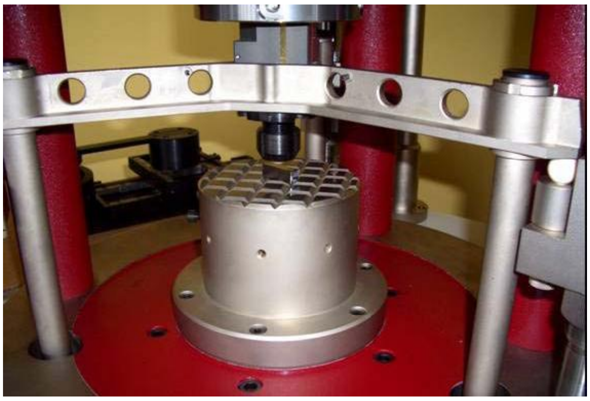

Fig. 5. Verification of the depth measuring system by comparison with gage blocks.

2.1.3. Stiffness

Stiffness of the machines have been verified measuring the deformation of the machine using a Rockwell test on the same indentation using a big ceramic ball indenter (19 mm diameter or 10 mm diameter) (fig. 6).

After 30 indentations, the maximum measured deformation between the preload levels (before and after the application of the full load) has been 0,03 µm, corresponding to 0,015 HRC points.

2.1.4. Indenters

All the indenters (Rockwell and Vickers) supplied with the primary machines have been calibrated at INRIM, both in direct (geometry) and indirect (by comparison of the INRIM national indenter) methods.

Calibration results are complied with the relevant standard specifications [15-17].

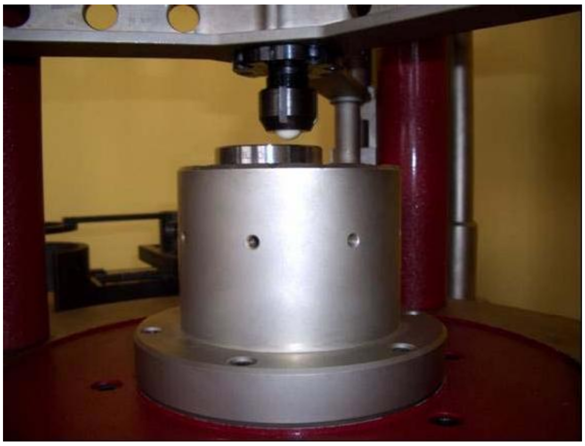

Figure 6: stiffness verification using a ceramic ball indenter (10mm of diameter)

2.2. Indirect verification

Indirect verification consists in the comparison of the hardness scales realized by the PHSMs with the Italian National Scales realized by the INRIM PHSM.

Machine parameters that can influence the measurement results [18], have been set according to the standard specifications [17]. Example of Rockwell 15N testing cycle output form the machines sensors is given in fig. 7. Before performing measurements, the machine load cells have been calibrated.

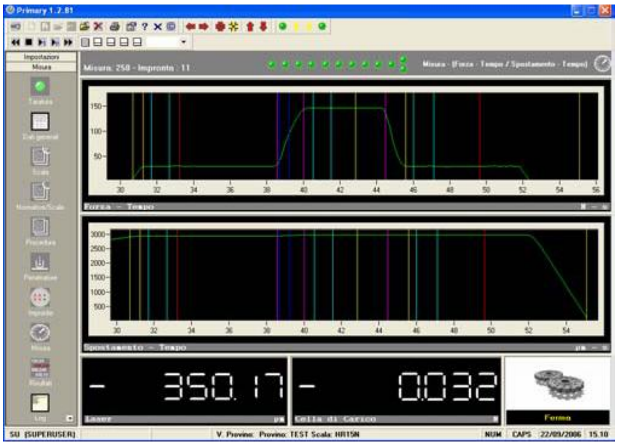

Fig. 7. Example of Rockwell 15N testing cycle

For the comparison, the machine effect has been separated by the indenter effect, using own and common indenters.

Taking into the account the differences of the acceleration due to gravity between LTF of Antegnate (Italy) and other NMIs locations that generate different forces in different sites, corrections have been calculate using sensitivity coefficients available in literature [12, 19, 20] or specifically calculated with experiments (for Rockwell N scales).

Example of typical results are presented in fig. 8.

As conclusion of the comparison, looking at the results obtained both with own and common indenters, the differences of the PHSMs are fully compatible [21].

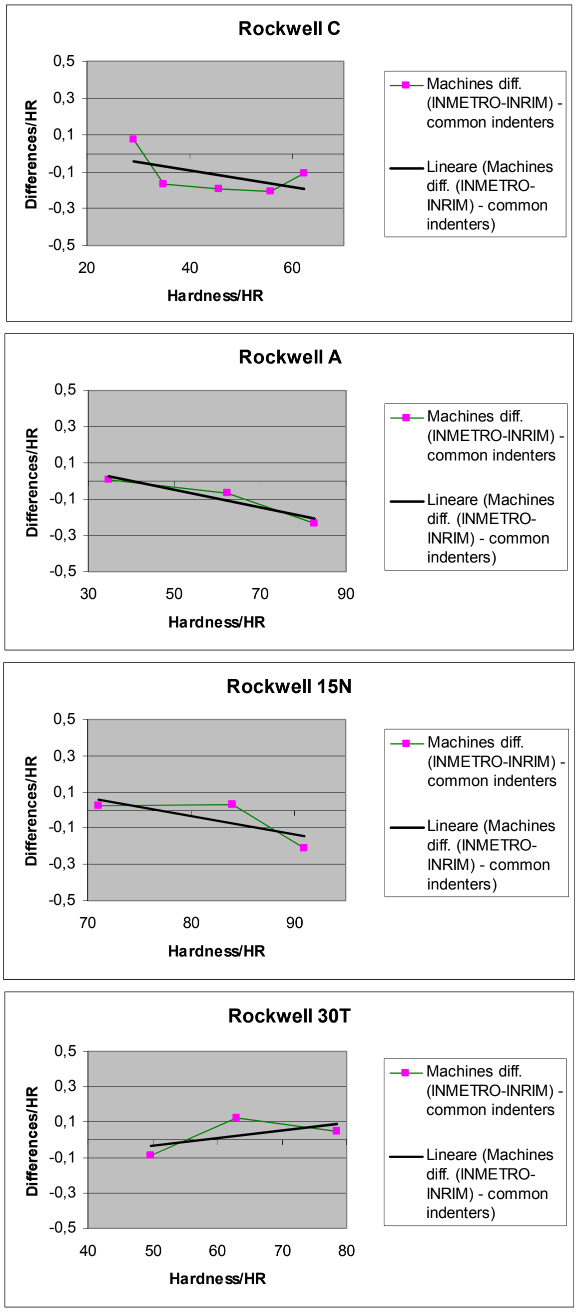

Fig. 8. Example of comparison results of PHSMs

3. VICKERS HARDNESS CALIBRATION MACHINE (VHCM)

The LTF VHCM is a dead weight machine with a lever amplification (20x). It has been supplied to NIM Hardness laboratory in Beijing, China.

The metrological characterization consists in the direct and indirect verification of the machine; in the direct verification, the main verified parameters are the forces generated by the VHCM and the geometry of the indenters. The indirect verification consists in the comparison of the hardness scales generated by the VHCM with the national scales maintained by INRIM.

Fig. 2. LTF Vickers Hardness Calibration machine for NIM (China)

3.1. Direct verification

3.1.1. Force

The VHCM generates forces by means of dead weighs and a lever amplification (20x) in the range from about 20N up to 1kN.

The specification of the VHCM on the force generation (maximum error of 0,1%) has been verified by mean of the direct verification of the forces generated by the machine thought a calibrated load cell with an adequate uncertainty (class 00 of ISO 376:2004) (fig. 9).

To guarantee that the VHCM works correctly, forces generated by the machine have been checked in different positions (low and high position) (fig. 10). The positions have been determined calculating the minimum and maximum penetration for each level of force for each HV scale realized by the machine. The displacement has been measured using the transducer on board of the machine.

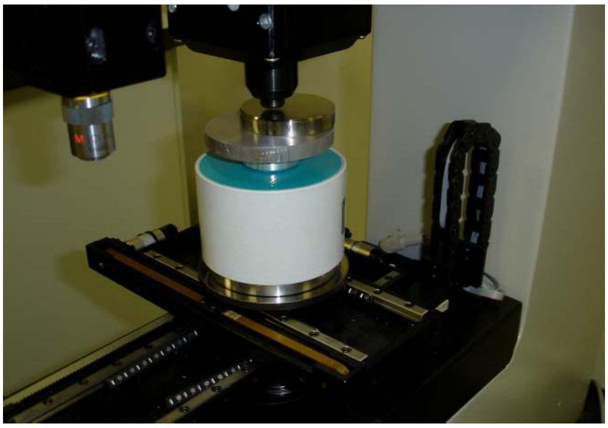

Fig. 9. Verification of the test forces with a load cell

Force measurements carried out at LTF laboratory have been corrected taking into the account the differences of the acceleration due to gravity between LTF-Antegnate in Italy and NIM-Beijing in China.

As it is possible to see in the figure, all forces generated by the VHCM are inside the tolerance of the ISO 6507-3 standard (± $1x10^{-3}$).

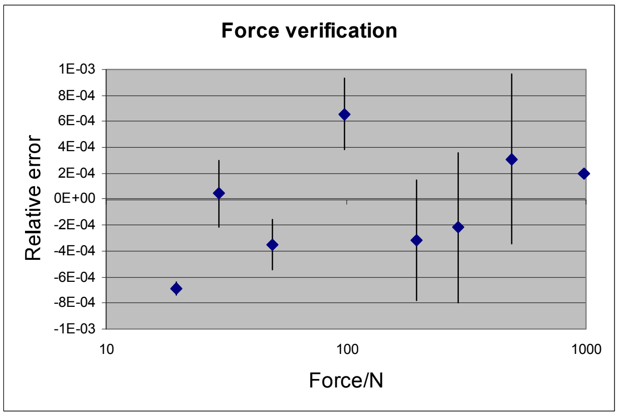

Fig. 10. Differences from the nominal force in the range from 20N up to 1kN. Dots represent mean values and error bars the maximum variation due to different height positions (corresponding to high and low hardness for each force-scale)

3.1.2. Vickers diamond indenter

Vickers diamond indenters have been calibrated by INRIM. All results fulfil the ISO 6507-3 specifications.

3.2. Indirect verification

Indirect verification consists in the comparison of the hardness scales realized by the VHCM with the Italian National Scales realized by the INRIM PHSM. Each machine has been used with its indenter.

All measurements were carried out complying ISO 6507-3 specifications.

Taking into the account the difference of the acceleration due to gravity between LTF-Antegnate (Italy) and NIM-Beijing (China) which generates, with same masses, different forces in the two different sites, corrections have been calculate using the sensitivity coefficients used in the last CIPM Vickers Key-Comparison and following indicated:

Results, with the application of the corrections, are shown in fig. 11.

The evaluation of uncertainty in measurements has been done following the procedure for the unified estimation of the measurement uncertainty adopted in the last CIPM Vickers Key-Comparison.

With this evaluations (differences and their uncertainties), the degree of equivalence (DoE) has been calculated. From the analysis of the DoE, all the differences are inside ±1, so measurements are compatible.

4. AUTOMATIC MEASURING SYSTEMS FOR VICKERS AND BRINELL INDENTATIONS (GAL-VISON)

The LTF Gal-Vision measuring systems for measuring automatically Vickers and Brinell indentations (fig. 12), derive from the measuring system developed at INRIM and based on the CNR patent algorithm [22-23].

The system allows the measurement of Vickers or Brinell indentations.

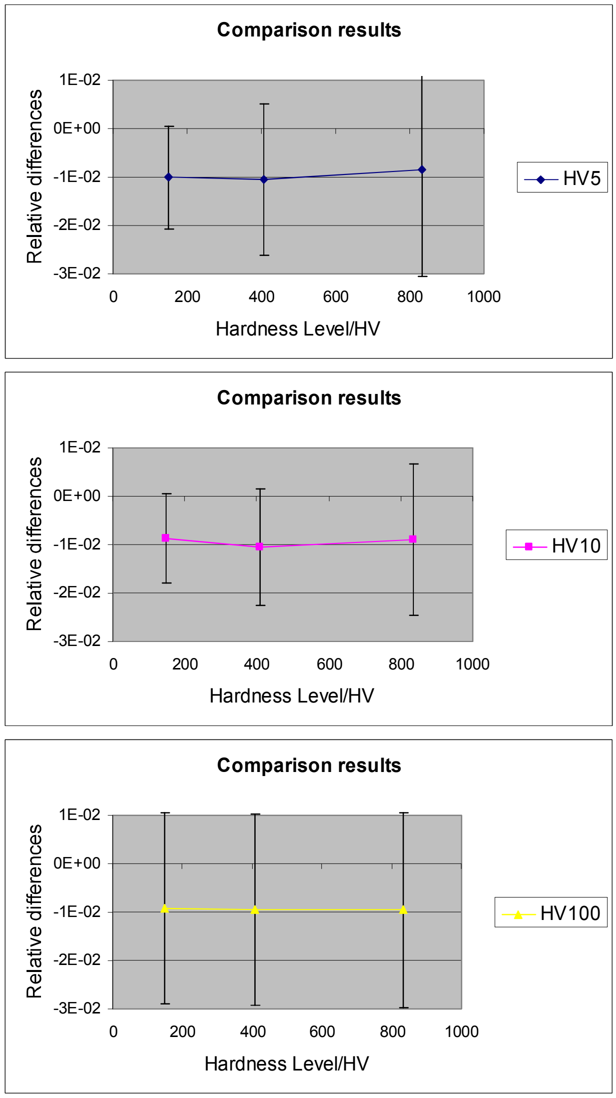

Fig. 11. Results of the comparison of the Vickers hardness scales realized by the LTF VHCM for NIM and INRIM PHSM

Fig. 12. Gal-Vision measuring system

On the base of effective sizes of each indentation and to get the best accuracy, the System can carry out the aforementioned measurements by two different methods:

• Direct method: it is the faster method and it is used when the size of the indentation is less of the visual field captured by the CCD; the System automatically measures diagonals or diameters of the indentation and calculates the corresponding hardness value. With the direct method, the system is able to measure Vickers (and Brinell) indentations with diagonal (or diameter) lengths starting from 0.02 mm (using 50x lens) up to 2.5 mm (using 2,5x lens) on the screen of the computer.

• Combined method: when the better accuracy is requested or when the size of the indentation is larger of the visual field due to choose lens, the System works with a combined method (reading by probe & CCD). The system moves automatically, by using the motorized stage along the X direction, the vertexes or points to be measured in the middle of visual field and acquires coordinates of the probe for that position. Rotating manually the indentation of 90° by a rotary stage and aligning manually the second diagonal or diameter to be measured, it is possible to measure the second diagonal or diameter with the same procedure. The System automatically calculates the value of the diagonal or the diameter, by the two positions of the vertex respect to the centre of the visual field and the difference of the two coordinates of the probe. The maximum stroke of the probe is 12 mm, so that it is possible to carry out even measurements on large Brinell indentations.

With the combined method, the system is able to measure Brinell indentations realized in all scales; starting from 9,807 N to 29420 N with ball indenters from 1 mm to 10 mm diameter, and Vickers indentations up to HV100 which may not be measured in the direct method. For both aforementioned methods, the accuracy and the uncertainty are according to the relevant ISO standard [15-16], and guaranteed by calibrated stage micrometers. The INRIM system uses a laser head instead of the probe, in order to have better accuracy in measurements.

The metrological characterization consists in the direct and indirect verification of measuring systems. In the direct verification, main parameters like pixel calibration, linear encoder accuracy, etc. are verified; in the indirect verification, a comparison of the whole performances of measuring systems are verified through a comparison with INRIM measuring systems.

4.1. Direct verification

The resolution of the linear encoder has been verified to be 0,1 µm as requested, in the most restrictive conditions, by the relevant standard [15-16].

The accuracy (error in tab. 2 of the relevant standard [16]) has been also verified through a comparison with calibrated stage micrometer. In tab. II, results of the verification are summarized and compared with standard specifications [16].

Gal-Vision measuring system has the possibility to perform the pixel calibration using, as reference standard, the liner encoder. To verify the calibration of the pixel dimensions, different indentation measurements with the two methods (direct and indirect) available in the Gal-Vision measuring system, have been carried out. With the comparison of measurement results obtained with these two different methods, it is possible to assure the corrected pixel calibration. For this purpose, “false” indentations of different dimensions have been used.

Considering that the influence of the algorithm used for the determination of the pixel position is included in worst results (±0,34 µm on about 500 µm diagonal length), its effect, in hardness points, is negligible compared with the uncertainty.

TABLE II. verification of the linear encoder

Diagonal length, d/mm	Maximum permissible error (ISO 6507-3)	Determined accuracy
d≤0,040	0,0002mm	$8x10^{-5}$ (<0,0001mm)
0,040 < d ≤ 0,200	0,5% of d	$8x10^{-5}$ (<0,01%)
d > 0,200	0,001mm	$8x10^{-5}$ (<0,0002mm)

4.2. Indirect verification

This verification has been carried out by means of a comparison between the LTF Gal-Vision measuring systems and the INRIM measuring system.

This comparison consists in the measurement of some Vickers indentations made on a reading block (fig. 13) and some “False” rhomboidal and spherical indentations made on a glass stage.

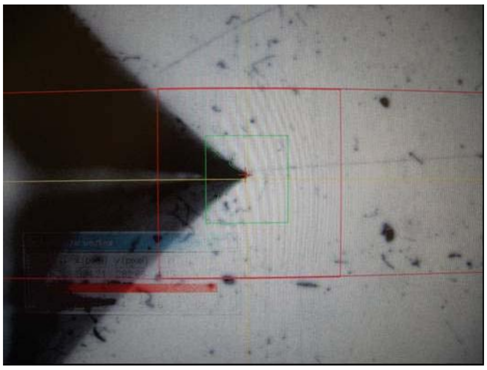

Fig. 13: fully automatic Vickers indentation measurement session.

Measurements have been carried out using different lenses with different Numerical Aperture (NA). Results are presented in fig. 14. Results show that all differences are inside $\pm 1x10^{-3}$ relative.

Typical results (fig. 14) show that, for the measurements on master indentations (Vickers and Brinell), the maximum difference is ±1,0 µm ($\pm 1,9x10^{-3}$ in relative). In the reading block measurements, the differences are typically within ±1,2 µm ($\pm 4,3x10^{-3}$ in relative).

This result, which include the effect of the different N.A. and diagonal measurement systems, is fully compatible with the contribution to the uncertainty due to this contribution, as calculated also in the last international comparisons.

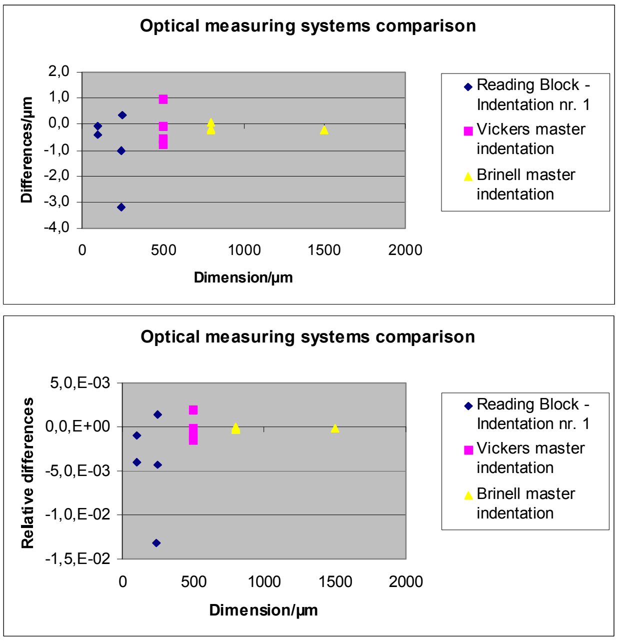

Fig. 14. Example of comparison results of LTF Gal-Vision and INRIM measuring systems

From the analysis and considering this effect compared with the hardness uncertainty (normally more than 1%), results are very satisfactory.

5. MEASURING SYSTEMS FOR THE GEOMETRIC CHARACTERIZATION OF ROCKWELL AND VICKERS INDENTERS (GAL-NDENT)

Gal-Indent measuring systems (fig. 16) has been developed by GALILEO-LTF in cooperation with INRIM [24-25].

Fig. 16. Gal-Indent measuring system

It perform the geometrical verification of the diamond tips of indenters for Rockwell, Rockwell superficial, Vickers and MicroVickers indenters. The system consists of:

- ♦ interferometric sine-bar for angular, straightness and flatness measurement.
- ♦ rotating table, for the verification of the spherical tip of Rockwell indenters.

The two instruments can be set up in one workstation, interfaced with the same computer for data analysis.

The metrological characterization consists in the direct and indirect verification of measuring systems. In the direct verification, main parameters like sine–bar geometry, angular and linear encoder accuracy, etc. are verified; in the indirect verification, a comparison of the whole performances of measuring systems are verified through a comparison with INRIM measuring system.

5.1. Direct verification

5.1.1. Geometrical checks

Gal-Indent Measuring Systems have been checked on their geometrical characteristics to assure the perfect alignment of their mechanical parts that are references for angle measurements.

The first check is the alignment of the plane of the granite base with the lens perpendicular axis. To perform this measurement, a calibrated gage block of 175 mm length has been used. As result, three interference fringes have been obtained in the interference image that corresponds to an angle of 0,06° in the orthogonal direction to the measurement angle direction (fig.17).

Fig. 17. Verification of the parallelism between the granite base and the lens perpendicular axis

The second check is the verification of the parallelism between the granite base and the stage axis. A calibrated square and a micrometric comparator has been used as shown in fig. 18. The variation from the parallelism has been found to be 2 µm on a run of 40 mm (0,002°).

On the other hand, a check on the orthogonal support of the linear encoder has shown a misalignment of 20µm on 100 mm (0,008°).

Fig. 18. Verification of the parallelism between the granite base and the stage axis

The linear encoder has been verified with comparison of calibrated gage block of 50 mm. An error of 5 µm on 50 mm has been found ($1x10^{-4}$).

The maximum error of repeatability and reproducibility of the measurement carried out with the linear encoder is within 38 µm.

Combining all the geometrical and measurement errors, the maximum error between the zero setting, corresponding to the position shown in fig. 19, and the measurement position is less than 0,02°.

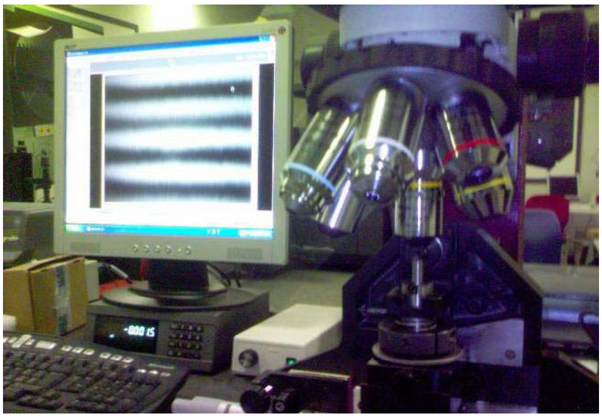

Fig. 19: position at zero

5.1.2. Angle measurement

There are two different kind of angle measurements:

- first, more metrologically relevant, is the measurement of the vertex angle of the Vickers and Rockwell indenters (nominally 136° and 120° respectively);
- second is the angle of the squareness of the quadrilateral intersection between the faces and a plane perpendicular to the axis of the Vickers diamond pyramid.

For the first angle measurements a sine-bar system is uaed and a interferometric lens is used as collimator. To evaluate the uncertainty of this kind of measuring system, the following evaluation has been done.

The light wavelength used to illuminate the indenter is 546 nm (declared value of the filter). Accepting a collimation error of the indenter surface inside 20 fringes, it corresponds about 11 µm error on the measurement of the sine-bar length. In addition, we can add the error of the linear encoder, estimated as maximum 5 µm. So the global error in the worst case is 16 µm that correspond, on a base length of 50 mm on 100 mm, about 0,011° of error. Considering the measurement uncertainty of the reference base length (100 mm) of 0,01 mm, the global error is 0,014°. This is much less than the declared value in the Gal-Indent technical specification (0,1° accuracy, 0,03° repeatability, 0,06° reproducibility). The influence of the value of the wavelength has been estimated and results shown that the global error does not change if the wavelength variations are inside 50 nm on 546 nm (about 10% variation). So, we can assume that the determination of the filter wavelength and its stability is surely inside the 10%.

The second angle measurements is performed by an angular encoder. A twelve faces calibrated polygon and an autocollimator has been used to verify the angular encoder. Four measuring cycles have been performed and results of this verification have shown a maximum expanded uncertainty (p=95%) on angle measurements of 0,02°.

Results of the verification show compatibility with the standard specifications (±0,2°) [15-16].

5.2. Indirect verification

Gal-Indent measuring system measures the geometrical characteristics of diamond pyramidal Vickers indenters or diamond conical Rockwell indenters. A comparison with the measuring system used at INRIM has been done to verify the whole performances of the Gal-Indent measuring system.

For this purpose, several Rockwell and Vickers indenters have been used for the comparison. All calibration results complied to the standard specifications [15-16].

Examples of differences obtained during the comparison are reported in tabs. III and IV.

TABLE III. Comparison between INRIM measuring system and LTF Gal-Indent for INMETRO measuring Vickers indenter

geometrical parameter	*Differences*	*Expanded uncertainty (p=95%)*	*Gal-Indent Specifications*	*ISO 5607-3 tolerance*
mean angle/°	-0,01	0,06	0,1	± 0,1°
length of line of conjunction/µm	0	0,28	0,25	<1
Planarity/µm	0	0,1	0,3	<0,3
diamond axis inclination/°	0,01	0,1	0,1	0,3
Angle of the quadrilateral (0°)/°	-0,06	0,08	-	± 0,4
Angle of the quadrilateral (90°)/°	-0,10	0,09	-	± 0,4
Angle of the quadrilateral (180°)/°	0,05	0,09	-	± 0,4
Angle of the quadrilateral (270°)/°	0,11	0,09	-	± 0,4

TABLE IV. Comparison between INRIM measuring system and LTF Gal-Indent for INMETRO measuring Rockwell indenter

geometrical parameter	*Differences*	*Expanded uncertainty (p=95%)*	*Gal-Indent Specifications*	*ISO 6507-3 tolerance*
Mean radius/µm	-0,60	2,3	3	5
Mean angle/°	0,06	0,09	0,1	± 0,1
diamond axis inclination/°	-0,02	0,06	0,1	0,3
straightness of generatrix/µm (L=0,4mm)	0,05	0,28	1	0,5

Repeatability and reproducibility measurements have been carried out to verify the Gal-Indent technical specifications, as well as the uncertainty of deviation of the tip profile. In table V the results are shown.

As we can see in the measurement results, the Gal-Indent performances have been verified to be inside its technical specifications.

TABLE V. measurements of repeatability and reproducibility of the LTF Gal-Indent measuring system for INMETRO

geometrical parameter	*repeatability*	*reproducibility*	*Expanded uncertainty (p=95%)*	*Gal-Indent Specifications*
Radius/µm	0,0	0,1	-	-
Angle/°	0,01	0,01	-	0,03/0,06
deviation of the tip profile/µm	0,1	0,1	0,4	0,5

3. CONCLUSION

The opportunity of new installations of Primary Hardness Standard machines and harness measuring system gave to INRIM and LTF the possibility to cooperate for new improvements of the apparatus. The activity of metrological characterization has demonstrated the perfect realization of the instrumentation and gave the possibility to make experience on the verification of Primary Hardness Standard machines and measuring systems in the field of harness at the level of NMIs laboratories.

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