XIX IMEKO World Congress
Fundamental and Applied Metrology
September 6−11, 2009, Lisbon, Portugal

NEW POSSIBILITIES IN THE GEOMETRICAL CALIBRATION OF DIAMOND INDENTERS

Alessandro Germak [1], *Claudio Origlia* [2]

[1] Istituto Nazionale di Ricerca Metrologica, INRIM, Torino, Italy, A.Germak@inrim.it
[2] Istituto Nazionale di Ricerca Metrologica, INRIM, Torino, Italy, C.Origlia@inrim.it

Abstract – The influence of Rockwell and Vickers diamond indenters geometry in hardness measurements justifies the strict specifications and tolerances in the relative standards. Many methods, and consequently dedicated instruments, have been developed to investigate the geometrical parameters in order to calibrate the diamond indenters. Most of them, using contact or contactless methods, can only investigate some profiles of the indenters; from these partial measurements the whole geometry is derived. Few other instruments can investigate the whole shape but, since they are custom-developed, costs and difficulties in the calibration of these instruments have limited their use.

A new method is proposed in this paper using a commercial and not dedicated instrument: a 3D measuring confocal microscope. This type of instrument have also the advantage that the whole geometry is analyzed with a consequently knowledge improvement in the indenter calibration.

In the paper the measurement method, the used instrument and its calibration to assure traceability to the length and angle standards are investigated.

Keywords: Hardness indenter, calibration, confocal microscopy

1. INTRODUCTION

The influence of Rockwell and Vickers diamond indenters geometry in hardness measurements is very well known ant it has been deeply investigated [1 to 6]. As result of these studies, the calibration of diamond indenters used for Rockwell and Vickers hardness measurements is one of the specifications in the relative international standards [7 to 10]. The tolerances vary if the indenter is used for industrial or calibration purpose, but they are always well defined.

Several instruments have been developed in the years to carry out the calibration of these types of indenters [11, 12]. Most of them have the following limits: i) partial investigation of some profiles of the indenter shape, ii) high cost due to the customized development, iii) difficulties in the instrument calibration to assure traceability to the unit standards.

In the recent years, a family of microscopes using the confocal technology have been developed and are now easily available on the market. One of the limits that they had in their first realizations was the impossibility to observe diamond surface. This limit is now overcome with new confocal technology and new types of sensors.

2. INSTRUMENT AND METHOD

The used instrument, available at INRIM laboratory, is a dual core 3D measuring microscope which combines both confocal and interferometry techniques, but only confocal lenses are used.

Confocal microscopy [13] is an optical imaging technique used to increase micrograph contrast and/or to reconstruct three-dimensional images by using a spatial pinhole to eliminate out-of-focus light or flare in specimens that are thicker than the focal plane [14].

The invention of the confocal microscope is usually attributed to Marvin Minsky, who produced a working microscope in 1955 [15]. The principle of confocal imaging advanced by Minsky, and patented in 1957, is employed in all modern confocal microscopes.

In a conventional (i.e., wide-field) fluorescence microscope, the entire specimen is flooded in light from a light source. Due to the conservation of light intensity transportation, all parts of the specimen throughout the optical path will be excited and the fluorescence detected by a photo detector or a camera. In contrast, a confocal microscope uses point illumination and a pinhole in an optically conjugate plane in front of the detector to eliminate out-of-focus information. Only the light within the focal plane can be detected, so the image quality is much better than that of wide-field images. As only one point is illuminated at a time in confocal microscopy, 2D or 3D imaging requires scanning over a regular raster (i.e. a rectangular pattern of parallel scanning lines) in the specimen. The thickness of the focal plane is defined mostly by the square of the numerical aperture of the objective lens, and also by the optical properties of the specimen and the ambient index of refraction. These microscopes also are able to see into the image by taking images at different depths.

This technique finds an optimum application observing and reconstructing the shape of diamond Rockwell and Vickers indenters.

The system is also equipped with a motor X-Y stages that permits to operate as a fully automated coordinated

ISBN 978-963-88410-0-1 © 2009 IMEKO

measurement system. With these features, extended profile and extended topography measurements can easily be acquired using the stitching algorithms included in the software. A closed-loop PZT assures a high-accuracy z-scan. It achieves sub-nanometer vertical resolution (up to 0,1 nm) and traceability to national standards.

Due to the versatility of the microscope, the measurement method is very simple. The indenter is located under the microscope lens (different magnifications and Numerical Apertures can be selected) and, after the setting of the measurement parameters, an automatic procedure is launched. It consists mainly in the link of several images by means of stitching algorithms.

The total measurement process needs a couple of hours but, since it is fully automatic, during this time interval no operation is necessary by the operator.

2.2. Instrument calibration

The first calibration of the instrument has been made by means of 2D calibrated gratings for the Z-axis. Similar tests of calibration have been carried out for the X-Y axis using 1D calibrated gratings [16, 17].

For this application, it is foreseen also to verify the instrument calibration directly by means of an angle standards and calibrated spheres (ruby spheres).

3. PRELIMINARY RESULTS

In fig. 1 an example of 3D image of the Rockwell indenter is presented. It is obtained using a 50x confocal objective with a N.A. of 0.55. It is composed by stitching 36 single images (6x6) each obtained with 10 µm Z-step scan (internal resolution of few nanometers). With this resolution the measuring time is about 2 hours but it is possible to increase the Z resolution up to 1 µm step, correspondent to a sub-nanometer internal resolution; at this maximum resolution the measuring time can considerably increase.

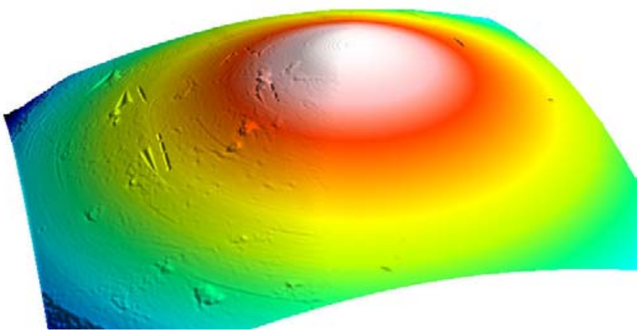

Fig. 1. Reconstruction of the tip geometry of a Rockwell indenter

Having the whole image of the indenter shape, with the software of the microscope is it possible to perform the measurement of the geometric parameters as required by the relative standards (angle of the cone, radius of the spherical tip, planarity of the generatrix of the cone, etc.). One of the most difficult to measure, that is probably the most influent in hardness measurements, is the radius of the spherical tip. As example is here reported the possibility to estimate this parameter.

The first step is the definition of the border limit between the spherical and the conical part of the indenter. At the present it is defined by the operator but it is foreseen the development of an automatic routine in the software for this analysis.

In fig. 2 the top view of the image of the spherical part of the indenter is shown.

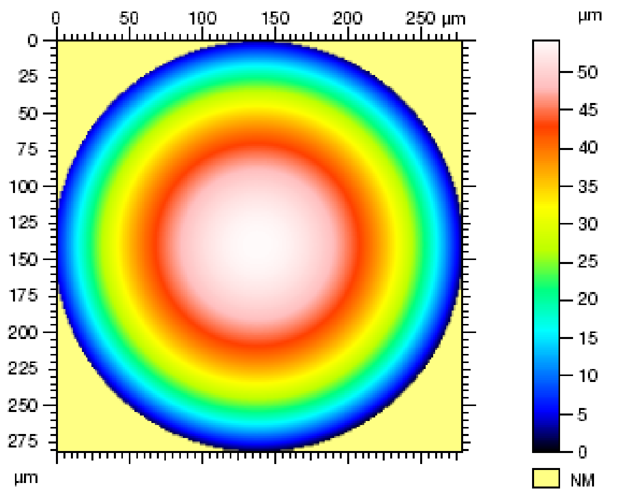

Fig. 2. Top view of the spherical tip of the Rockwell indenter

From this image, the software can automatically interpolate a theoretical sphere, giving the result of the radius of the sphere (and its standard deviation). In the example, the interpolated sphere has a radius of 214 µm.

It is also very useful to have, as result of the interpolation process, the picture representing the residuals of the interpolation that are the deviations from the interpolating sphere (fig. 3).

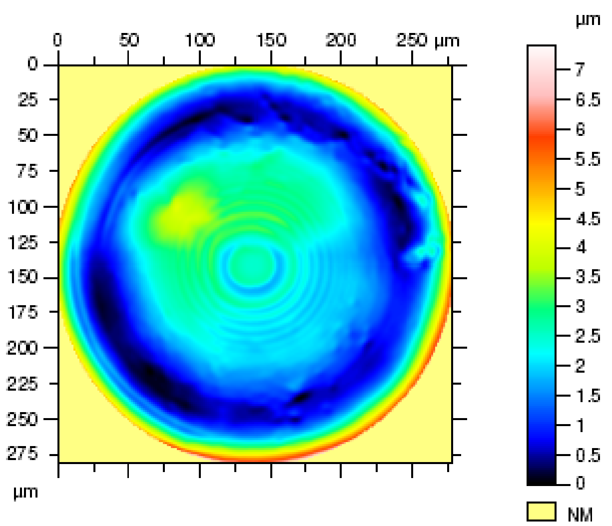

Fig. 3. Deviation from the interpolating sphere (214 µm)

From that picture is quite easy to see not only the maximum variations (in this case 7 µm, corresponding to

±3,5 µm from the mean value of 214 µm), but also as the deviations are distributed in order to have also information about the quality of the indenter geometry.

3.1. Comparison with other methods

In order to validate the measurement method, a comparison with another system has been carried out.

At IMRIM hardness laboratory a specific instrument has been developed for the calibration of hardness indenters. It consists in a sine-bar system with interferometric microscope used for the angle measurements, and a rotating table with an LVDT transducer (used as comparator with a ruby sphere standard) for the radius measurements [18].

The comparison consists in the analysis of the differences of the measuring values obtained with the two different measuring systems on four different cross sections (at the present only the radius of the spherical tip has been analysed).

From the image obtained by the confocal microscope it is very easy to extract a line profile of selected sections of the spherical tip of the indenter (fig. 4).

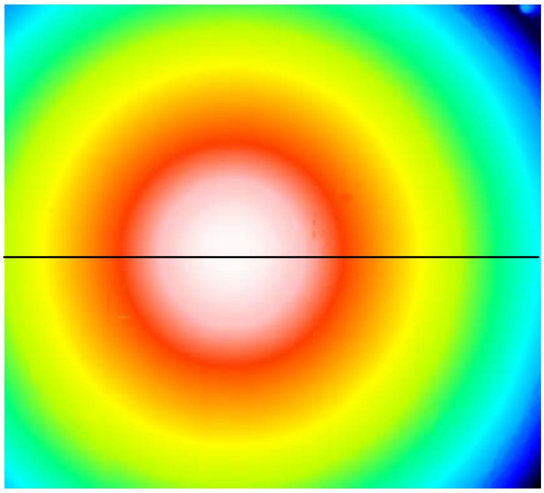

Fig. 4. Extracted profile (Rockwell indenter)

From this profile it is possible to zoom on the spherical part as shown in fig. 5.

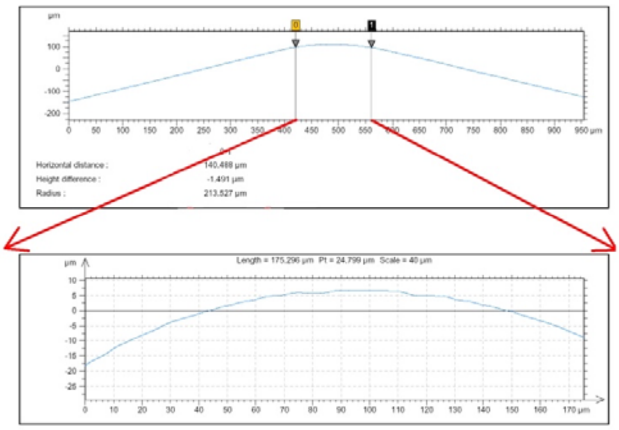

Fig. 5. Profile section with zoom on spherical tip

Using the tools available in the software of the confocal microscope, the radius of the interpolated circle is calculated as 214,12 µm. Since for the comparison with the other above described instrument it needs the calculation of 8 measurement sections, the image acquired with the confocal microscope has been elaborated in with the same procedure as made for the other instrument. An example of the elaboration is shown in fig. 6.

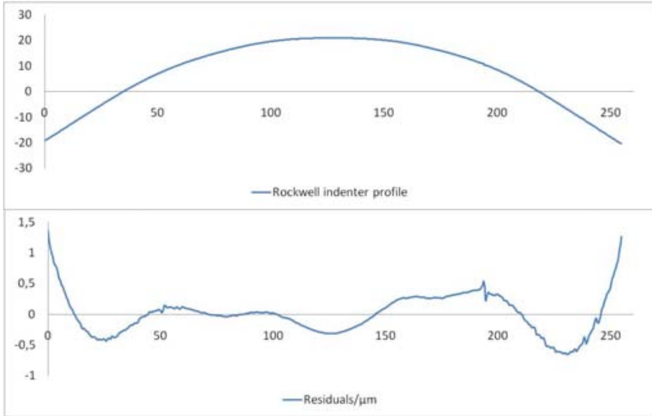

Fig. 6. Profile section analysis of the Rockwell indenter tip and residuals from spherical interpolation (212,04 µm)

From the comparison of the results of the two different systems based on the measurement of eight angular sections equally observed, differences of the order of 1÷2 µm have been measured. These differences are fully compatible with the expanded uncertainties of the measuring systems that are in the order of 2÷3 µm.

However, the work on the uncertainty estimation of the measurements obtained with the confocal microscope should be better developed and it is probably possible to obtain better uncertainties varying the acquisition parameters and carrying out a more accurate instrument calibration.

Similar investigation has been carried out with a Vickers indenter.

Also in this case two different measurement methods can be applied: profile measurement and full surface analysis.

From the image obtained by the confocal microscope (fig. 6) a line profile of selected sections of the pyramidal tip of the indenter has been extracted (fig. 7) and analysed (fig. 8).

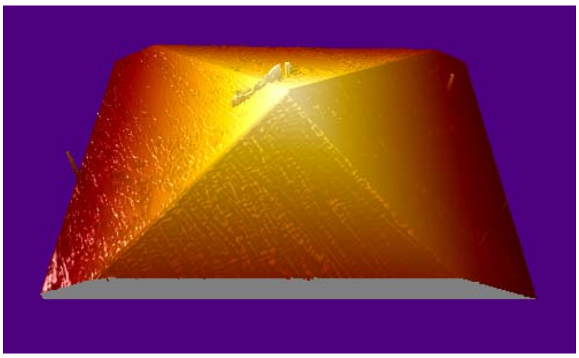

Fig. 6. A 3D image of Vickers indenter

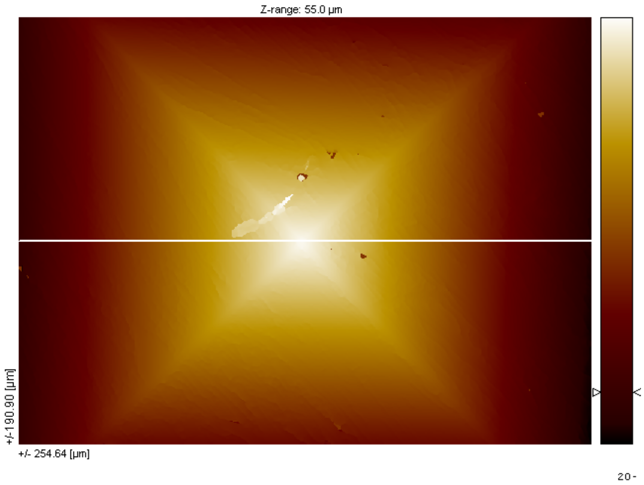

Fig. 7. Extracted profile (Vickers indenter)

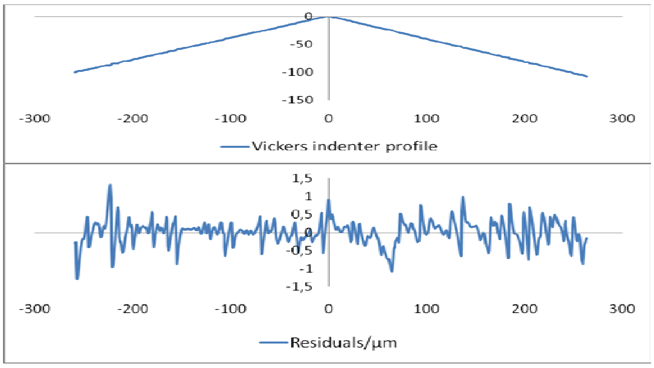

Fig. 8. Profile section analysis of Vickers indenter with residuals from linear interpolation (135,97°)

Using the tools available in the software of the confocal microscope, the angle between two plane surfaces interpolating opposite faces of the Vickers indenter is calculated as 135,99°.

The data calibration obtained with the other instrument give a result of (136,00±0,03)°, fully compatible with the measurements obtained with the confocal microscope.

4. CONCLUSIONS

The technique of the confocal microscopy has been demonstrated to the able to satisfy the necessity of measurements in the hardness diamond indenters calibration.

Using this type of microscope more complete information about the geometry of indenters are obtained compared with stylus-type instruments.

This preliminary work intends only to investigate this technique but more intensive and deep work is necessary to estimate all the geometrical parameters involved in the indenter calibration and to estimate correctly the uncertainty of measurement. However, from the preliminary results, it seems that uncertainties at the level of the state of the art of this kind of measurements, or better, can be easily reached.

REFERENCES

[1] Marriner R. S., Wood J. G., “Investigation into the Measurement and Performance of Rockwell C Diamond Indenters”, Metallurgia, August 1967, 87, 87-90.

[2] Barbato, G., Desogus, S., Levi, R., "The Meaning of the Geometry of Rockwell Indenters," Rapp. Tec. Int., Sept. 1978, R128.

[3] F. Petik, “Factors Influencing Hardness Measurement (A Systematic Survey of Research Results)”, Bureau International de Metrologie Legale ,Paris, 1983 (restricted distribution).

[4] Barbato, G; Petic, F, “Metrological Involvement in the Definition and Dissemination of Hardness Scales”, proceedings of XIII IMEKO, Torino, 1994, 1, 761-766.

[5] Barbato, G., Galetto, M., Germak, A., Mazzoleni, F., “Influence of the indenter shape in Rockwell hardness test”, Proc. of the HARDMEKO ‘98, Sept. 21-23, Beijing, China, 1998, p.53-60.

[6] J. Song, S. Low and L. Ma, “Form error and hardness performance of rockwell diamond indenters”, Proceedings of 16th World Congress of International Measurement Confederation (IMEKO XVI), Vienna, Austria, September 01, 2000, 6 pp.

[7] ISO 6507-2 “Metallic materials — Vickers hardness test — Part 2: Verification and calibration of testing machines”

[8] ISO 6507-3 “Metallic materials — Vickers hardness test — Part 3: Calibration of reference blocks”

[9] ISO 6508-2 “Metallic materials — Rockwell hardness test — Part 2: Verification and calibration of testing machines”

[10] ISO 6508-3 “Metallic materials — Rockwell hardness test — Part 3: Calibration of reference blocks”

[11] A. Germak, K. Herrmann, G.Dai, Z. Li, “Development of calibration methods for hardness”, VDI-Berichte Nr. 1948, pp. 13-26, 2006.

[12] Satoshi Takagi, Kazuyuki Kamijo, Takashi Usuda, Haruo Kawachi, Kazutomi Hanaki, “Wide-range verification of the geometry of vickers diamond indenters”, XVIII IMEKO WORLD CONGRESS, Metrology for a Sustainable Development, September, 17 – 22, 2006, Rio de Janeiro, Brazil

[13] D. Semwogerere, ER Weeks, "Confocal Microscopy", Encyclopedia of Biomaterials and Biomedical Engineering, Taylor & Francis, 2005, pp. 1-10.

[14] *Handbook of Biological Confocal Microscopy* (3rd Ed.). Berlin: Springer. Pawley JB (editor), 2006.

[15] Marvin Minsky, "Memoir on Inventing the Confocal Scanning Microscope," Scanning, vol.10, pp. 128-138, 1988

[16] R. Bellotti, G.B. Picotto, M. Pisani, F. Pollastri, M. Pometto, “Calibration of 1D, 2D gratings and linescales”, Workshop NanoMetrology 2007 – Metrology for Nanotechnology, Torino, June 2007

[17] M. Bisi, E. Massa, A. Pasquini, G. B. Picotto, M. Pisani, “Nanometrology at the IMGC. Nanoscale calibration standard and methods”. Ed. G. Wilkening, L. Koenders, WILEY-VCH, Weinheim, pp.22-36, 2005.

[18] A. Liguori, A. Germak, G. Gori, E. Messina, “Galindent: the reference metrological system for the verification of the geometrical characteristics of Rockwell and Vickers indenters”, VDI-Berichte, vol. 1685, pp. 365-371.Sept. 2002.