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

A SIMPLE MATHEMATICAL METHOD USED TO DESCRIBE THE INDENTER TIP AREA FUNCTION

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Abstract – The method called instrumented indentation test (IIT) emerged as a technique capable to characterise several materials in nanoscale, like nanostructural material, thin film, ceramic material, etc., independently of hardness.

The acceptation of this new technique requires, of course, reliable test results where, one of the way to obtain this confidence is to knowing the sources of the testing error in order to minimise them.

This work has as main objective to present a simple methodology to get one of these sources by a mathematical computation to determining the geometry of the Berkovich diamond indenter used in the IIT focusing on the very tip of this indenter up to 200nm height by adjusting the curves that better describe this range.

Keywords: Instrumented indentation test, Indenter area function, Nanoscale.

1. INTRODUCTION

The measurements of the conventional indentation hardness, Brinell (HB), Rockwell (HR), Vickers (HV) and Knoop (HK) are known and their methods were normalised more than 50 years ago. The results of HB, HV and HK are numbers that represent the size of the residual impressions on the sample surface after the indentation and the results of HR are numbers related to the indentation depth. Face to scientific and technological demands, noticed in the superficial engineering and in the last decades in nanotechnology field, different methods were developed for the characterisation of hardness and other mechanical material properties in micro and nanometer scales.

The method known as "Instrumented indentation test for hardness and materials parameters" [1] or simply IIT, is based on the use of diamond indenters to indent the sample surface, with forces and indentation depths measured and presented simultaneously through a graphic by means of a curve Force versus indentation depth (*Fxh*) in the screen of a computer. With this technique, is possible to characterise the plastic and elastic behaviour of a material in nanometer regions (<200 nm), where it has been frequently the only one which it makes possible the research of nanostructural material, thin film, ceramic material, etc., independently of hardness.

In order to obtain the indentation hardness from this technique, some standards and devices are used for the correct measurement. Amongst the indenters, a triangular based diamond indenter (Berkovich) is one of the most common. Nevertheless, the determination of the true contact area of this indenter through the regular procedure [2] could find some complexity due to the geometry deviation of the very tip of this indenter. In such case, this could make the indentation hardness measurement, in nanometer scale, an arduous task.

In order to well characterise the materials used in IIT, is fundamental the knowledge of the indenter geometry and consequently the area of contact between the indenter and a sample during the measurement. This is because the indenters are not perfectly sharp and the theoretical shape of its tip does not represent a reality in the nanoscale. This shape can be affected manly by the lapidating process of the diamond and by its crystal anisotropy - that is the case of the Berkovich (pyramidal of triangular base) indenter used in this work.

The geometry of the indenter can be measured either direct or indirectly. The direct method can be done by Scanning Electro Microscopy (SEM) or Scanning Force Microscopy (SFM) [2], however, they are time consuming (indenter metalizing, indenter assembly on the device, indenter tip axis alignment), they are dependent of the image resolution and besides, it's necessary to have an operator with high expertise. The indirect method although is more easy and practical. It can be done from empirical testing in homogeneous materials with knows indentation hardness and modulus using several ranges of force and displacement to determine a function to describe the indenter geometry.

This function is related with the projected area of contact, $A_p(h_c)$, and the indentation contact, h_c, of the indenter from its tip and it is normally determined by the best fit coefficient. This process can include a calculation considering several indentations into a considered certified reference material (CRM) and its data of force, displacement and stiffness.

This article aim to present a simple mathematical computation to determine the area function of the Berkovich indenter used to describe the contact area between the indenter tip and the sample in nanometer scale.

2. MATERIALS AND METHODS

In this study, were used three kinds of considered CRMs, the SiO_2, the BK7 and the N-LAK33 [3]. These three materials were also used as the samples to be measured. The equipment used to do the nanoindentation tests was a Nanoindenter XP (MTS System Corporation, Oak Ridge, Tennessee, USA).

According to the ISO14577-2 standard [4] the determination of the area function could be done by a polynomial adjustment (1) based on the ideal geometry of a Berkovich indenter (2).

$$A_p(h_c) = C_0 \cdot h_c^2 + C_1 \cdot h_c + C_2 \cdot h_c^{1/2} + C_3 \cdot h_c^{1/4} + C_4 \cdot h_c^{1/8} + \ldots + C_n \cdot h_c^{1/n} \quad (1)$$

$$A_p(h_c) = C_0 \cdot h_c^2 \quad (2)$$

The initial term of (1) and (2), C_0, is knowing as the leader, and it could be written as a constant 24,5, where it is normally determined by a trigonometric relation of the triangular based pyramid. The other terms, C_1, C_2, ..., C_n, of the (1) describes the geometric deviation of the indenter tip.

2.1. Proposal of new methodology

From the metrological point of view, the determination of the curve that describes the indenter geometry could be equal to a conventional calibration by comparison, where a true value [5] could be used to obtain an “error” value for a standard. Then this “error” could enter in the measured values budget during the quantity dissemination.

Bearing in mind this mode of comparison for the IIT, a “reference” curve obtained from the indentation hardness true values of a reference material could be used to determine the hardness of the remaining materials (samples). Then the determination of this curve could be done by using (2) and (3), where the indentation hardness (H_{IT}) and the maximum applied force (F_{max}) have to be known in order to determine the h_c.

$$H_{IT} = \frac{F_{\max}}{A_p(h_c)} \quad (3)$$

Based on (2), which refers to a perfect geometry, the constant C_0 complies with a perfect pyramid of triangular base. So, this constant could be replaced by a calculated curves (4) or (5) through an interpolation of h_c values obtained in each nanoindentation test [6].

$$A_p(h_c) = \left(0{,}1385 \cdot h_c^{-0{,}348}\right) \cdot h_c^2 \quad (4)$$

$$A_p(h_c) = \left(2 \cdot 10^{15} \cdot h_c^2 - 5 \cdot 10^8 \cdot h_c + 67{,}889\right) \cdot h_c^2 \quad (5)$$

The equation 4 was obtained through a power interpolation, using the theoretical constants X, where X was got by using (3) modified, and the value of F_{max} and H_{IT} of each test for each sample.

In equation 5, the term in parentheses represents the curve obtained using the same methodology but with a polynomial adjustment.

The adjust could be changed according to the behaviour of the achieved constant for each CRM, where the hardness values could be determine either by a polynomial of second degree or by a power adjust. The method used in this work to obtain the curves was the Least Square Method because it normally provides a best fit from experimental tests [7].

Once the interpolate curve is done, the polynomial and exponential adjustments could be carried out.

Applying the proposed method, comparisons were made with all material. These materials were used either as a reference or as the samples to simulating a calibration. The values obtained were compared with the results from the software Analyst®, used by the nanoindenter XP-MTS®.

The criteria used to evaluate the proposed methodology were the relative error (E_R) (6) between the true value (V_T) and the measured value (V_M) obtained from the proposed methodology.

$$E_R = \frac{V_M - V_T}{V_T} \cdot 100 \quad (6)$$

3. RESULTS

In order to determine the indenter area function using the proposed methodology, it was considered the indentation contact, h_c, previous performed on the samples SiO_2, BK7 and N-LAK33 [3].

In each one of the three tests performed, the values of h_r (intersection point of the tangent to the force removal curve at F_{max} with the indentation depth-axis) were extracted and then used to calculate the values for h_c according to the ISO 14577-1 procedure [8].

The table 1 shows the results of h_c, in three levels of maximum force (Test A, Test B and Test C), obtained for the N-LAK33 CRM, where the hardness true value for the this material (9,8 GPa) were inserted into (3) to obtain the constants “X” given on the table 2 and used to calculate the indenter area.

Table 1. h_c results of the three tests performed, using the N-LAK33 as CRM.

Test	h_r (nm)	h_{max} (nm)	F_{max} (mN)	h_c (nm)
Test A	39,906	59,673	0,983	44,848
Test B	61,479	87,886	1,967	68,080
Test C	108,632	149,133	4,913	118,757

Table 2. Constants *X* calculated by the proposed method from IIT results for the N-LAK33 as CRM.

Test force	h_c (m)	*Constant X*
Test A - 1mN	0,000000045	49,86239
Test B - 2mN	0,000000068	43,29723
Test C - 5mN	0,000000119	35,54414

From these new constant values, table 2, an interpolate curve, Fig. 1, using a polynomial adjustment was described for a Berkovich indenter tip area.

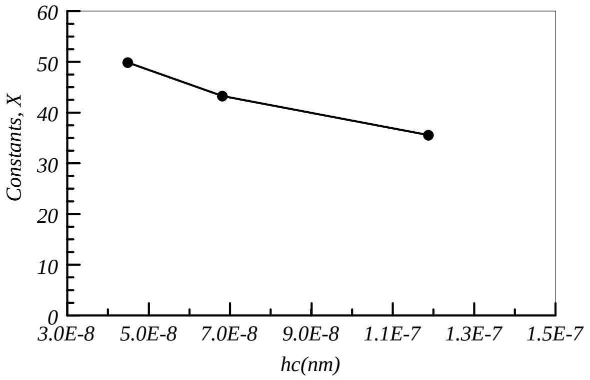

Fig. 1. Interpolated curve of the reference material, N-LAK33, from the polynomial adjustment.

The table 3 shows the results of indentation hardness (software and V_M), calculated for SiO2 and BK7 samples, using the N-LAK33 as the reference material and the indenter geometry extracted by (5) throughout a polynomial adjustment.

Table 3. Measured results (V_M) with a polynomial adjustment, machine software and hardness errors, using the N-LAK33 as CRM.

SiO_2	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error* (%)
Test A	8,271	11,426	10,100	24,196
Test B	8,143	10,833	11,492	17,750
Test C	7,833	10,497	14,853	14,098
BK7	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error* (%)
Test A	6,204	10,883	27,862	26,547
Test B	7,339	9,361	14,662	8,849
Test C	6,672	8,736	22,414	1,581

The absolute and relative errors indicated in table 3 were obtained from the comparison between the true hardness values of the samples SiO2 and BK7, (9,2 GPa and 8,6 GPa respectively) and from other two values obtained by the nanoindenter software and by the proposed methodology with a polynomial adjustment.

The relative error considered acceptable in this work was when the values obtained with new proposed method presented a smaller value compared with those calculated from the conventional method.

In the same way the table 4 is showing the results of absolute and relative error, by using the N-LAK33 as CRM but, in this time using a power adjustment.

Table 4. Measured results (V_M) with a power adjustment, machine software and hardness errors, using the N-LAK33 as CRM.

SiO2	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error*
Test A	8,232	11,426	10,520	24,196
Test B	8,113	10,833	11,812	17,750
Test C	8,351	10,497	9,223	14,098
BK7	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error*
Test A	6,182	10,883	28,120	26,547
Test B	7,325	9,361	14,830	8,849
Test C	7,385	8,736	14,127	1,581

The same procedure used to obtain the results of the tables 1 and 2 and then the tables 3 and 4 was the one used to carry out the comparison of the other two cited materials (SiO2 and BK7). In one moment it was used the SiO2 as CRM to construct the tables 5 and 6 and then the tables 7 and 8 and in another one, it was used the BK7as CRM to provide the values given on the tables 9, 10 and 11.

The polynomial adjustment obtained with the reference material SiO2 is represented by (7) and the power adjustment is represented by (8).

$$A_p(h_c) = \left(2\cdot 10^{15}\cdot h_c^2 - 4\cdot 10^8\cdot h_c + 61{,}061\right)\cdot h_c^2 \qquad (7)$$

$$A_p(h_c) = \left(0{,}1514\cdot h_c^{-0{,}3357}\right)\cdot h_c^2 \qquad (8)$$

Test	*hr (nm)*	*hmax (nm)*	*Fmax (mN)*	*hc (nm)*
Test A	41,349	75,105	0,983	49,788
Test B	74,196	127,089	2,456	87,419
Test C	111,971	187,109	4,916	130,756

From the results of the table 5 and (3) it was possible to calculate the constants *X* showed on the table 6.

Table 6. Constants *X* calculated by the proposed method from IIT results for the SiO2 as CRM.

Test force	*hc* (m)	Constant *X*
Test A - 1mN	0,000000050	43,110
Test B - 2mN	0,000000087	34,929
Test C - 5mN	0,000000131	31,253

Figure 2 is showing an interpolated curve by using the polynomial adjustment and the constants of the table 6 to described indenter tip area of a Berkovich indenter.

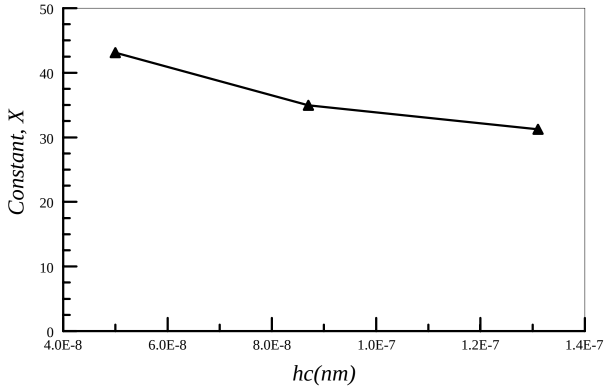

Fig. 2. Interpolated curve of the reference material, SiO2, from the polynomial adjustment.

The table 7 and 8 shows the results of indentation hardness (software and V_M), calculated for BK7 and N-LAK33 samples, using the SiO2 as the reference material by applying the polynomial and power adjustment respectively.

Table 7. Measured results (V_M) with a polynomial adjustment, machine software and hardness errors, using the SiO2 as CRM.

BK7	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error* (%)
Test A	6,431	10,883	25,222	26,547
Test B	6,898	9,361	19,787	8,849
Test C	5,582	8,736	35,088	1,581
N-LAK33	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error* (%)
Test A	10,365	14,026	5,765	43,122
Test B	9,845	13,166	0,461	34,347
Test C	8,340	12,012	14,894	22,571

Table 8. Measured results (V_M) with a power adjustment, machine software and hardness errors, using the SiO2 as CRM.

BK7	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error* (%)
Test A	6,951	10,883	19,170	26,547
Test B	8,177	9,361	4,918	8,849
Test C	8,203	8,736	4,621	1,581
N-LAK33	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error* (%)
Test A	11,017	14,026	12,418	43,122
Test B	11,005	13,166	12,300	34,347
Test C	10,890	12,012	11,123	22,571

In this study, by using the BK7 as CRM and the behavior of the constant *X* (table 10), it was possible to determine and validate the results of the indenter tip geometry only through a polynomial adjustment.

Table 9. h_c results of the three tests performed, using the BK7 as CRM.

Test	*hr (nm)*	*hmax (nm)*	*Fmax (mN)*	*hc (nm)*
Test A	45,019	71,067	0,781	51,531
Test B	82,643	124,069	2,455	92,999
Test C	126,489	183,695	4,9119	140,790

Table 10. Constants *X* calculated by the proposed method from IIT results for the BK7 as CRM.

Test force	*hc* (m)	Constant *X*
Test A - 1mN	0,000000052	34,21807
Test B - 2mN	0,000000093	33,01470
Test C - 5mN	0,000000141	28,81381

From the constant *X* of the table 10, it was possible to determine the curve, Fig. 3, that describe the indenter tip geometry from the use of (9) and the BK7 data as reference material.

$$A_p(h_c) = \left(-7 \cdot 10^{14} \cdot h_c^2 + 7 \cdot 10^{7} \cdot h_c + 32{,}552\right) \cdot h_c^2 \qquad (9)$$

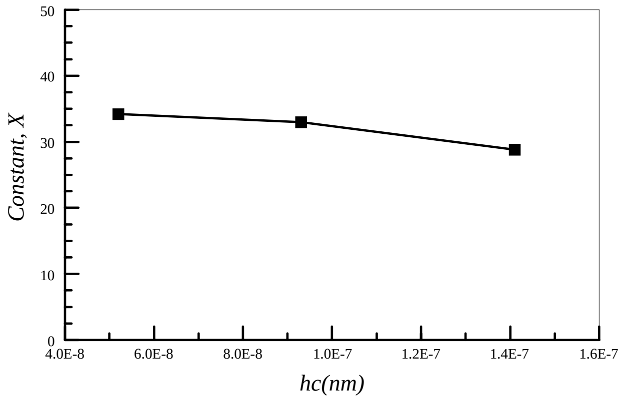

Fig. 3. Interpolated curve of the reference material, BK7, from the polynomial adjustment.

The following table 11 is presenting the measurement result obtained from the samples SiO2 and NLAK33 through theirs comparison with the BK7 CRM.

Table 11. Measured results (V_M) with a polynomial adjustment, machine software and hardness errors, using the BK7 as CRM.

SiO2	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error* (%)
Test A	11,562	11,426	25,677	24,196
Test B	9,644	10,833	4,822	17,750
Test C	9,669	10,497	5,099	14,098
NLAK33	V_M (GPa)	*Software* (GPa)	V_M *error* (%)	*Software error* (%)
Test A	14,253	14,026	45,442	43,122
Test B	12,453	13,166	27,071	34,347
Test C	11,239	12,012	14,685	22,571

4. DISCUSSION

The development of new processes of hardness characterisation and new instrumented indentation devices claims to the existence of trial researches in order to make the instrumented indentation hardness determination more easier and friendly in order to have a confident technique as well as hardness results with warranty quality for the users.

Having this issue in mind, this work has presented a new methodology to determine the indenter geometry tip used in the indentation hardness in nanoscale (h<200 nm) by applying a curve adjustment.

With this methodology, it was possible to get the geometry tip in a simple mode with reliability, where one could use a known reference hardness values to measure the indentation hardness in the nanoscale for other materials that have close hardness values.

From the results obtained in this study of comparison, it was possible to notice that the methodology proposed is satisfactory, since among the thirty comparisons performed, eighteen tests presented a relative error lower than those measured by the conventional method (ISO14577-2), it means, 60% of the results had a positive assessment.

It was also possible to observe that the SiO_2 material provided a better performance as a reference material among the three materials used. Maybe this could be done due to the fact that this material presented an intermediate true value of the other tow materials.

ACKNOWLEDGMENTS

The author 1 thanks to the National Institute of Standards and Technology (NIST/USA), where the nanoindentation test were performed. The author 2 thanks to Prof. Augusto César de Castro Barbosa from Universidade do Estado do Rio de Janeiro – UERJ for his support in the mathematical computation.

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