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ScienceDirect
Journal of the European Ceramic Society 27 (2007) 1905–1911
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Comparison of instrumented Knoop and Vickers hardness measurements on various soft materials and hard ceramics

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Received 5 January 2006; received in revised form 6 June 2006; accepted 23 June 2006
Available online 18 September 2006

Abstract

Vickers and Knoop hardness measurements performed on various ceramics (hard metals) and light alloy materials (soft metals) are compared. The results show that the Knoop hardness number is, in general, lower than the Vickers hardness number for the highest values of hardness, and this behaviour is reversed when the hardness values are low. This change in values, which occur at 8 GPa, has no real physical meaning and, therefore, it is difficult to interpret such behaviour in terms of the elasto-plastic deformation around the indent such as sinking-in, piling-up, and bulging of the indent faces, phenomena which take place during indentation or after the withdrawal of the indenter.

Prior to interpreting the hardness difference, it is very important to consider the same area in the hardness calculations. That is why we have compared the available hardness data obtained from the literature and recalculated them by considering the projected and true areas of the contact. If the objective is to compare the two hardness numbers, it seems more suitable to consider the true area of contact, procedure which will provide a Vickers hardness number higher than the Knoop hardness number all over the range of the hardness values.
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Keywords: Mechanical properties; Hardness; Nitrides; Carbides

1. Introduction

The hardness of a material is defined as the resistance to plastic deformation usually when the indentation test is carried out. The principle of indentation consists in applying a given load and, subsequently, measuring the dimensions of the residual impression left in the material once the indenter has been withdrawn. Hardness of the material is then defined as the ratio between the indentation load and a parameter representative of the area of the residual impression, depending on the shape of the indenter and the method employed for the hardness calculation.

For the Vickers hardness test, the indenter is a square-based pyramid for which the angle, ψ, between the two opposite sides is equal to 136°. The representative area corresponds to the true area of contact between the pyramid and the material at the maximum load of indentation. By means of simple geometrical considerations, the contact area may be expressed as a function of the diagonal of the indent. The Vickers hardness number (VHN) generally used is then calculated using the following formula:

$$\mathrm{VHN} = \frac{P}{A_{\mathrm{TAC}}} = \frac{P}{d^2/2 \cdot \sin(\psi/2)} \left(= 1.8544\frac{P}{d^2}\right) \quad (1)$$

where VHN is expressed in MPa, if P the applied load is in N and d is the diagonal of the indent in mm. A_{TAC} represents the true area of contact.

The Knoop hardness test used a lozenge-based pyramid with the angle θ between the two opposite faces being 172°5 and the angle φ between the other two being 130°. Calculation of the Knoop hardness number considers the projected area of contact in the plane of the material. The projected area is calculated using the length of the indent by knowing the theoretical relationship between the length and the width of the impression. The Knoop hardness number (KHN) is calculated as follows:

$$\mathrm{KHN} = \frac{P}{A_{\mathrm{PAC}}} = \frac{P}{L^2\,\mathrm{tg}(\varphi/2)/2\,\mathrm{tg}(\theta/2)} \left(= 14.229\frac{P}{L^2}\right) \quad (2)$$

where KHN is expressed in MPa, if P the applied load is in N and L is the large diagonal of the indent in mm. A_{PAC} represents the projected area of contact.

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doi:10.1016/j.jeurceramsoc.2006.06.011

In the majority of the hardness studies, different authors compare the Vickers and Knoop hardness measurements by using these two forms of calculations for the hardness numbers. We will show above that this approach leads to a wrong result, which is characterized by an inexplicable change of behaviour at a given value of the hardness.

2. Analysis of hardness data obtained from literature

Prior to the discussion related to various hardness measurements performed on hard ceramics, previous comments should be made on the validity of the experimental data. With the aim to normalize the hardness measurements, some rules and procedures were set and listed in several international standards such as the European draft standard ENV 843-4,[3] the National Institute for Standards and Technology[4] and the Fraunhofer-Institute for Ceramic Technologies and Sintered Materials.[5] Since these standards allow to perform appropriate measurement routines, the hardness values that were carefully measured under these recommendations could provide a valuable data base which could be used in order to compare the influence of the two indenter's geometries on the hardness numbers. Moreover, since each laboratory carried out the hardness measurements by employing the two indenter's geometries on the same sample, the comparison between the two hardness numbers, HV1 and HK2, is rendered possible and the discussion is then consistent for all data available from different laboratories.

Therefore, in the present work, we used the data provided by Ullner et al.,[1,2] which have tested several typical commercial ceramics, mainly silicon nitride, silicon carbide and aluminium oxide, by employing ENV 843-4 standard. However, Ullner et al.[1,2] raised the problem of the reproducibility between hardness measurements carried out in different laboratories and have concluded that the magnitude of standard deviations are independent of both hardness technique employed and laboratory were the measurements were carried out, and are, probably, associated with the variation of the microstructural characteristics of the material, i.e. porosity, grain orientation and grain size.

All the hardness values, HV1 and HK2, obtained from Ullner et al.[1] are summarised in Table 1. In order to complete a series of examples for hard metals, we have added the results presented by Gong et al.[6] on various ceramics based on silicon nitrides containing a different amounts of yttrium and lanthanum oxides. All the specimens were subjected to Vickers and Knoop indentations under the same applied load of 2.45 N, which was sufficiently high to avoid indentation size effect (ISE).[7] Additional general information of ISE allowing the hypothesis mentioned above are given, for example, in the extended review published by Cheng and Cheng.[8]

The hardness values obtained by Gong et al.[6] are the average results from of 10 indentations tests for each type of indenter and the reported error related to the diagonal measurement was of ±0.5 μm. Additionally, these authors have also published part of the experimental data obtained by Mukhopadhyay et al.[9] for seven sintered silicon nitride ceramics and five liquid-phase sintered SiAlON. Fig. 1 presents these two sets of experimental data reported by Gong et al.[6] which follow nearly the same trend.

To take into consideration much lower hardness values, we are considering the hardness results reported by Shaw et al.[10] for four metallic alloys. The average hardness values are in the range of 1–2 GPa and have been obtained by applying loads ranging between 0.1 and 10 N. Table 2 presents these results, which are the average of at least 10 indentations/applied load. Taking into account the hardness values obtained as a function of the applied load, we could consider that this material does not present an ISE. Fig. 2 represents all the hardness data as a function of the applied load indicating the correspondence between the two hardness numbers. In this figure, it is shown without ambiguity that the KHN values are lower than VHN values for those materials, which exhibited high hardness (Fig. 2a). At the contrary, for the materials that have low hardness, the KHN values are superior to VHN values (Fig. 2b). In a first

Table 1
Data related to the hardness numbers for some ceramic materials reported by Ullner et al.[1]

Code	Source/material code	Type	HV1[a]	HK2[a]
A	NIST/SRM 2830	HPSN	15.80	14.30
C	IKTS	GPSN	14.70	13.50
D	IKTS	GPSN	14.80	13.80
E	Tenmat/Nitrasil R	RBSN	10.20	9.40
F	Lucas-Cookson/Syalon 201	SiAlON	16.00	14.10
G	IKTS	LPS-SiC	25.60	19.60
H	CERAMTEC/CD	SiC	26.50	17.20
I	CERAMTEC/RK	Al_2O_3	18.90	16.10
J	IKTS	Al_2O_3	21.20	17.30
K	Morgan Matroc/VITOX (white)	Al_2O_3	19.90	17.10
M	Morgan Matroc/VITOX (white) + tempered	Al_2O_3	18.00	15.70
R	IKTS	GPSN	15.00	13.80
S	IKTS	LPS-SiC	24.90	20.20
T	IKTS	Al_2O_3	20.70	17.40
U = E	Tenmat/Nitrasil R	RBSN	10.20	9.30
V	IKTS	SiC	25.10	21.60
W = I	CERAMTEC/RK	Al_2O_3	18.90	16.10
X4, X5, X6, X7	IKTS	HPSN	14.60–17.70	13.50–15.30

[a] Hardness values are given in GPa.

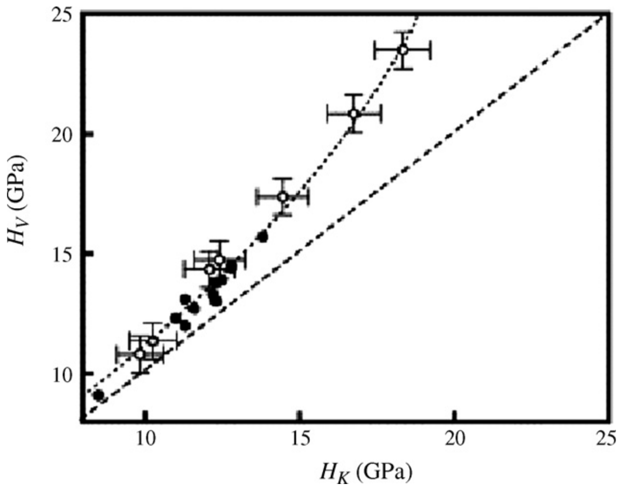

Fig. 1. Comparison between VHN (H_V) and KHN (H_K) using data reported by Gong et al.[6]

approximation, Mukhopadhyay et al.[9] have suggested that HVN and HKN numbers could be correlated by a calibration factor. In confirmation to the first conclusion of Gong et al.,[6] it is obvious from Fig. 2 that a straight line cannot represent the experimental data, especially for high hardness values. Then, we propose to fit the experimental data over all the range of values, i.e. from 0 to 30 GPa, by using a second order polynomial expression and considering that the two hardness numbers are equals when the value of the hardness tends to 0. In these conditions, the fitting curve leads to the following relationship:

$$\mathrm{KHN} = 1.1053\mathrm{VHN} - 0.0134\mathrm{VHN}^2 \quad (3)$$

From a mathematical point of view, it is possible to solve Eq. (3) in order to find the value for which KHN is equal to VHN in addition to the point when the hardness tends to 0 GPa. The solution of this equation gives a hardness value, so-called limit value, HL (i.e. HL = 7.9 GPa) for which the following conditions are verified:

$$\text{if VHN} < \text{HL then, KHN} > \text{VHN} \quad \text{and}$$
$$\text{if VHN} > \text{HL then, KHN} < \text{VHN} \quad (4)$$

The difference between the Knoop and Vickers hardness numbers has been subject to some interesting discussion in the past, but it was mainly centered on the high values of the hardness (>HL). That is why, the use of the general explanations for lower values of hardness leads to erroneous interpretations.

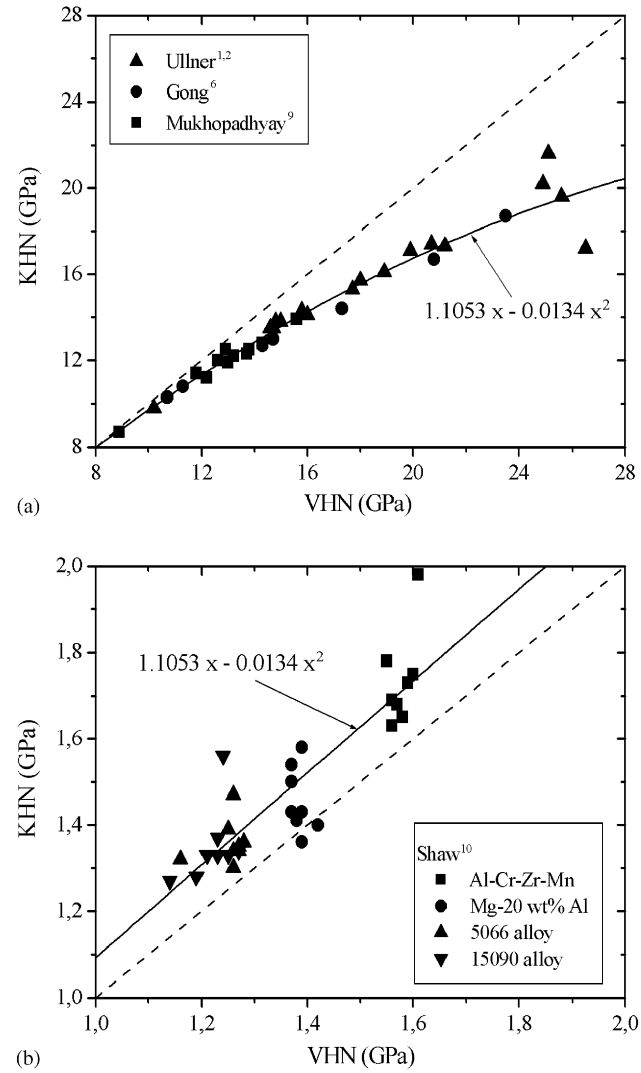

Fig. 2. Comparison between KHN and VHN including all tested materials (a) for high hardness values and (b) for low hardness values.

For example, Shaw et al.,[10] Atkinson et al.[11,12] and Shi and Atkinson[13] have tried to explain the difference of the two hardness numbers, Knoop and Vickers, through the friction phe-

Table 2
Vickers (VHN) and Knoop (KHN) microhardness measurements as a function of loads for four extruded light alloy materials made from rapidly solidified particulate, reported by Shaw et al.[10]

Load (N)	Al–Cr–Zr–Mn		Mg–20 wt.% 11		Allied 5066		Allied 5090	
	VHN	KHN	VHN	KHN	VHN	KHN	VHN	KHN
0.1	1.61	1.98	1.37	1.50	1.26	1.47	1.24	1.56
0.25	1.55	1.78	1.39	1.43	1.16	1.32	1.14	1.27
0.50	1.60	1.75	1.39	1.58	1.26	1.30	1.19	1.28
1	1.59	1.73	1.42	1.40	1.28	1.36	1.21	1.33
2	1.57	1.68	1.38	1.41	1.27	1.34	1.23	1.33
3	1.58	1.65	1.37	1.54	1.27	1.35	1.27	1.34
5	1.56	1.63	1.39	1.36	1.25	1.39	1.23	1.37
10	1.56	1.69	1.37	1.43	1.26	1.34	1.25	1.33

nomenon which takes place between the material and the indenter, stating that friction was dependent of the indenter geometry. To test this hypothesis they have carried out the indentations by using a lubricant and it was found that its presence contributed to an important decrease in the value of the hardness numbers and, consequently, to an increase in the value corresponding to the indent diagonal, for all the range of the applied load. Nevertheless, in presence of the lubricant, the hardness is always load dependant and the Knoop and Vickers hardness numbers are always different fact, which implies that this difference could not be only attributed to the friction phenomenon.

In addition, it has been reported by Lawn and Howes[14] and Marshall et al.[15] that an elastic recovery may occur after the indentation load is removed as the result of the mismatch between the plastic zone beneath the indentation and the surrounding elastic deformed material. Due to the specific shape of the indenter, the elastic recovery during Vickers indentation is rather different from that corresponding to Knoop indentation. In particular, Lawn and Howes[14] put forward a proposal that the elastic recovery occurs along the depth of the impression but the length of the indent diagonal is nearly unchanged for the Vickers indentation.

On the other hand, Marshall et al.[15] have observed that the length of the minor diagonal of the Knoop indent is often shorter than those expected by taking into account the geometrical considerations of the indenter. This phenomenon is also attributed to the elastic recovery. Therefore, Marshall et al.,[15] Blau[16] and Lima et al.[17] have proposed to express the ratio between the width, w, and the length, L, of the Knoop impression as a function of the ratio between the hardness, KHN, and the Young modulus, E, of the material by the following relationship:

$$\left.\frac{w}{L}\right|_{\text{measured}} = \left.\frac{w}{L}\right|_{\text{theoritical}} - 0.45\frac{\text{KHN}}{E} \quad (5)$$

where w/L theoretical ratio is equal to 0.1406, calculated from the geometrical characteristics of the Knoop indenter.

On the base of the above-mentioned assumptions,[15–17] Gong et al.[6] explains that the difference between the measured values of VHN and KHN may be attributed to the difference between the degree of the elastic recovery in both cases neglecting the fact that the plastic zone may be different. Nevertheless, Gong clearly shows a strong correlation between the hardness ratio VHN/KHN and the w/L ratio for their studied materials. The following relation proposed by Gong et al.[6] is obtained by a linear regression:

$$\left.\frac{w}{L}\right|_{\text{measured}} = 0.1908 - 0.0595\left(\frac{\text{VHN}}{\text{KHN}}\right) \quad (6)$$

Gong indicated that VHN/KHN = 1 gives a w/L value of 0.1313 being close to the theoretical value of 0.1406. But, if we consider the hardness value, HL, equal to 7.9 GPa calculated here above for which VHN is equal to KHN (or VHN/KHN = 1), that means that no elastic deformation would take place for a material whose hardness is equal to 7.9 GPa. Then Eq. (6) is obviously not applicable. Moreover, this relation is not accurate when VHN/KHN < 1 (i.e. in the range 0–7.9 GPa) because the measured ratio w/L became higher than the theoretical ratio. It is obvious that this result has no physical meaning because it involves an elastic collapse. In conclusion, this equation seems to be only applicable for hardness values higher than 7.9 GPa. It becomes then difficult to explain why such relationship cannot be applied over all the range of hardness data. In any case, comparison of the two hardness numbers should take into account the global deformation at the neighbourhood of the indent like piling-up, sinking-in, elastic recovery of the indenter faces, elastic withdrawal of the top of the residual indent, which all are dependent on the shape of the indenter. This calculation of hardness is more or less attempted by analysing the load–depth curve as proposed by Oliver and Pharr.[18] In the same way, in order to compare Knoop and Vickers hardness numbers, Zhang and Sakai[19] suggested a concept of an equivalent cone providing an efficient analytical method by which all the indentation behaviours for the Vickers and Knoop indenters were described in a unified manner. To establish their theory, Zhang and Sakai discussed about the ratio between the total penetration depth and the contact depth induced at the maximum load obtained by analysing the load–depth curve. But, if the instrumented hardness measurements are employed, it is not yet possible to reach to its ratio with the exception of the case when 3D optical analysis is used, which drastically increases the simplicity of such well-known mechanical test. Consequently, one of the main objectives of the present work is to perform a correlation between Vickers and Knoop hardness numbers obtained by discrete application of loads, which should be firstly easily applied and secondly valid for the entire range of hardness values from a physical point of view. To accomplish this objective, we suggest taking into account the same definition of the representative area of the indent into the well-admitted hardness calculations.

3. Reinterpretation of the hardness data

In their work, Zhang and Sakai[19] emphasized that the Vickers hardness and the Knoop hardness should be calculated in the same way, i.e. by considering the projected area of the residual indent. In this condition, Eq. (1) used to compute the Vickers hardness number should be changed to

$$\text{VHN}_{\text{PAC}} = \frac{P}{A_{\text{PAC}}} = 2\frac{P}{d^2} \quad (7)$$

where A_{PAC} indicates the projected area of contact and VHN_{PAC} should be now compared to the Knoop hardness number calculated from Eq. (2).

All the Vickers hardness values indicated in Fig. 2 could be recalculated by multiplying VHN by 1.0785, which represents the ratio between the coefficients of Eq. (7) and that corresponding to the standard Vickers hardness definition, i.e. 2 and 1.8544, respectively, in order to obtain VHN_{PAC}. Fig. 3 shows the Knoop hardness number (KHN) as a function of the Vickers hardness number calculated by using the projected area (VHN_{PAC}) with Eq. (7). In this situation, the fitting polynomial becomes:

$$\text{KHN} = 1.0223\text{VHN}_{\text{PAC}} - 0.0114\text{VHN}_{\text{PAC}}^2 \quad (8)$$

For which, it will exist always a limit hardness value, HL_{PAC} (i.e. $\text{HL}_{\text{PAC}} = 2$ GPa).

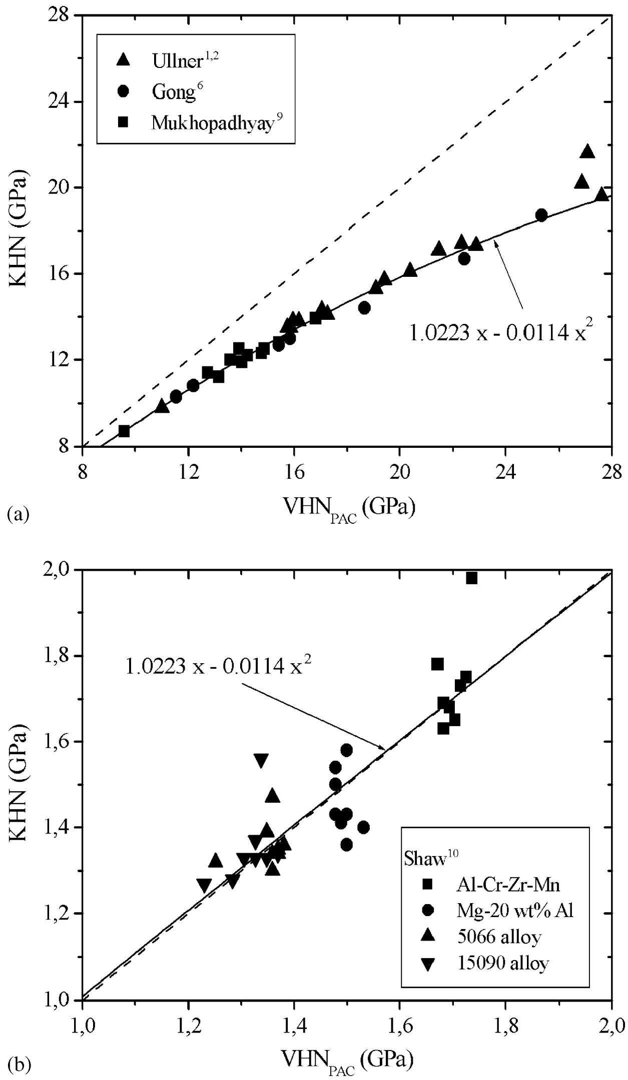

Fig. 3. Comparison between KHN and VHN_{PAC} including all tested materials (a) for high hardness values and (b) for low hardness values.

This result is not yet fully satisfactory since HL_{PAC} is not equal to 0 GPa. To improve this result, some authors like Saha and Nix[20,21] and Kese et al.,[22] have recently discussed about the possibility to take into account in the hardness calculation the true contact area determined directly from the measured contact stiffness. Nevertheless, for instrumented hardness tests, it is not possible to calculate the true area of contact after the withdrawal of the indenter. That is why, no further proposals were made in this direction, since this technique will be difficult to implement. To avoid such consideration about the deformation of the indent faces, the true area of contact could be computed by means of simple geometrical considerations of the pyramid, i.e. by considering the indenter still inside the material. For that purpose, the Knoop hardness calculation by means of Eq. (2) should be modified by taking into account the true area of contact, A_{TAC}, which could be deduced from the triangle delimited by a, b and c as indicated in Fig. 4.

The two angles $\varphi/2$ and $\theta/2$ necessary for the calculation take the values corresponding to 65° and 86°15′, respectively. The

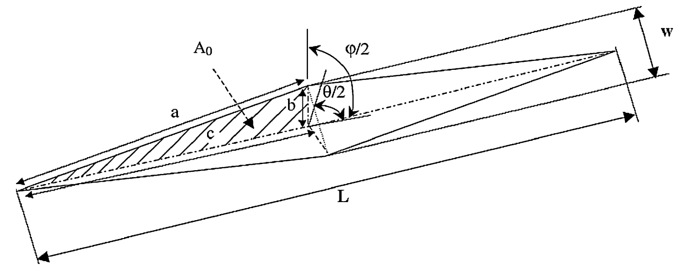

Fig. 4. Geometrical parameters used for the calculation of the true area of contact.

area, A_0, of each of the four triangles shown in Fig. 4 can be calculated by

$$A_0 = \sqrt{s(s-a)(s-b)(s-c)} \quad \text{with } s = \frac{a+b+c}{2} \tag{9}$$

a, b and c could be expressed as a function of L and of the two angles gives:

$$a = \frac{L}{2}\left(1 + \frac{\mathrm{tg}^2\varphi}{\mathrm{tg}^2\theta}\right)^{1/2}, \qquad b = \frac{L}{2}\left(\frac{1}{\cos\varphi\,\mathrm{tg}\,\theta}\right),$$
$$c = \frac{L}{2\sin\theta} \tag{10}$$

and their values could be substituted in Eq. (9), which will allow the general definition of the hardness. In these conditions, a new coefficient is obtained for the Knoop hardness, which may be now written as KHN_{TAC} where the TAC index indicates that the true area of contact was taken into account:

$$KHN_{TAC} = \frac{P}{A_{TAC}} = 12.873\frac{P}{L^2} \quad \text{with } A_{TAC} = 4A_0 \tag{11}$$

which differs only by its numerical coefficient, 12.873, instead of 14.229, from the standard definition, and should be now compared to the Vickers hardness number calculated from relation (1).

Using Eq. (11), it is now possible to represent the Knoop hardness number KHN_{TAC} as a function of the Vickers hardness (VHN). All the values of Knoop hardness indicated in Fig. 2 could be recalculated by multiplying KHN by 0.9047, which represents the ratio between the coefficients of Eq. (11) and that of standard Knoop hardness definition, respectively (i.e. 12.873/14.229), in order to obtain KHN_{TAC}. Fig. 5 shows the Knoop hardness (KHN_{TAC}) calculated for all the experimental results as a function of the Vickers hardness number. In this situation, the following relationship gives the fitting polynomial of the experimental data shown in Fig. 5:

$$KHN_{TAC} = VHN - 0.012VHN^2 \tag{12}$$

The main important result is that the coefficient in front of VHN term in Eq. (12) is equal to 1. This coefficient is calculated from the ratio between the two coefficients corresponding to the standard equation for computing (KHN) and the proposed equation for the Knoop hardness number (KHN_{TAC}), respectively, the later being equal to (14.229/12.873) = 1.1053. It is important to notice that this value is the same as that corresponding to the

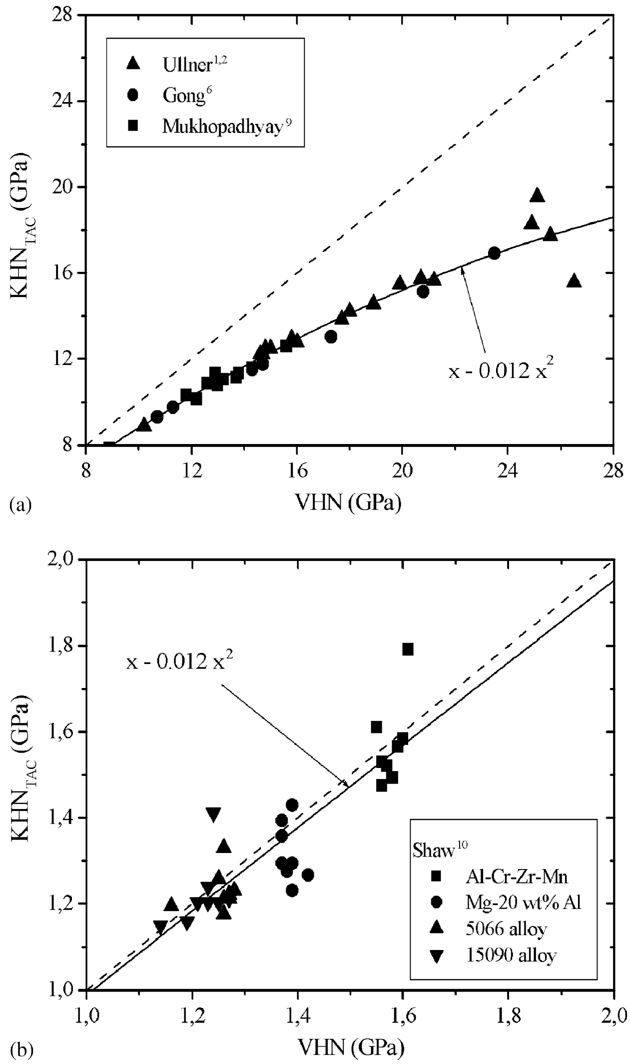

Fig. 5. Comparison between KHN_{TAC} and VHN including all tested materials (a) for high hardness values and (b) for low hardness values.

value of the coefficient of VHN in Eq. (3). Now, the two hardness numbers are equals only when the hardness value tends to 0 and, as a consequence, no surprising change of the behaviour will occur over the entire range of the hardness data. Indeed, as it can be seen from Fig. 5, the KHN_{TAC} values are below the VHN values over the whole range of hardness with an increase of the difference between them.

It is now possible to simply write the relative variation of the hardness:

$$\frac{\mathrm{VHN}-\mathrm{KHN_{TAC}}}{\mathrm{VHN}} = 0.012\mathrm{VHN} \quad \text{or} \quad \frac{\Delta H}{H} = \frac{\mathrm{VHN}}{83} \tag{13}$$

In these conditions, Eq. (6) proposed by Gong et al.[6] is rendered suitable for its application for any hardness value and allows removing all ambiguity related to the fact that Knoop hardness will equal Vickers hardness at 7.9 or 2 GPa, the later value being obtained if in calculation of the Vickers hardness the projected area of contact was taken into consideration. This conclusion allows us to rewrite the relation of Gong et al. as follows:

$$\left.\frac{w}{L}\right|_{\text{measured}} = 0.1908 - 0.0658\left(\frac{\mathrm{VHN}}{\mathrm{KHN_{TAC}}}\right) \tag{14}$$

In addition, the use of Eq. (5) is still possible if we adjust the coefficient of 0.45–0.5 (equal to product between 0.45 and 1.1053) as follows:

$$\left.\frac{w}{L}\right|_{\text{measured}} = \left.\frac{w}{L}\right|_{\text{theoritical}} - \frac{1}{2}\frac{\mathrm{KHN_{TAC}}}{E} \tag{15}$$

If we base our discussion on the comparison of the hardness values obtained, it seems that hardness must be calculated by considering the true area of contact. This is achieved without difficulties for any instrumented hardness test if the calculation is performed with the contact areas obtained under load for the two types of indenters. Nevertheless, in a recent study of the depth–load registration curve, Li et al.[23] suggested two methods for indentation hardness calculations based on the difference introduced by the contact area under load and the residual projected area of indentation after complete unloading. This approach allows these authors to discuss more accurately on the sinking-in, piling-up and elastic recovery. The value obtained for the hardness number by using this original approach is, however, quite different from that obtained by employing Oliver and Pharr's methodology.

Independently of such result, the load–depth registrations offer a large possibility of hardness calculations according to the area or the indentation depth considered into the hardness calculation. Then, for the instrumented hardness tests, the problem remains entire on the choice of the indentation area. Here, we propose a simple way for the hardness calculations by considering the true area of contact, procedure which will allow a valid comparison between the hardness numbers obtained when using Vickers or Knoop indenters. However, additional work is necessary in order to be able to link the difference between Vickers and Knoop hardness numbers to phenomena such as sinking-in, piling-up and elastic recovery.

4. Conclusions

The Knoop and Vickers hardness numbers have been expressed on the same basis by taking into account the true area of contact between the indenter and the material, which allows a better understanding of the difference between the two. In this condition, Knoop hardness number is always lower than the Vickers hardness number. This important result allows us to render appropriate the relationship proposed by Gong et al.,[6] which links the hardness ratio to the deformation of the Knoop indenter all over the range of the hardness data. The as-defined Knoop hardness in the present work does not impede the use of the relationship proposed by Blau[16] and Lima et al.[17] if the above-mentioned adjustment is performed.

Acknowledgements

This work has been conducted in the framework of a Postgraduation Cooperative Programme (PCP), between the University of Science and Technology in Lille and the Universidad Central de Venezuela, supported by both the French Ministry of Foreign Affairs (MAE) and Youth, National Education and Research (MJENR) and the Venezuelan National Fund for Science, Technology and Innovation (FONACIT).

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