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STUDY OF THE INFLUENCE OF THE ELASTIC RECOVERY DURING CONVENTIONAL KNOOP INDENTATION TESTS ON CERAMICS

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Abstract

It is generally observed in the literature, that the Knoop hardness number is lower than the Vickers hardness number, in particular for ceramics. This difference is due, on the one hand to the elastic recovery occurring during the removal of the Knoop indenter, and on the other hand to the difference between the representatives areas considered in each hardness calculation formula.
In this work, conventional hardness tests with Knoop and Vickers indenters were carried out in order to show how Knoop hardness test can give the same hardness number obtained by Vickers hardness test. This is obtained when Knoop hardness number is calculated based on the residual plastically deformed area whether projected or true. Complementary hardness data obtained from the literature were used in this work in order to validate the method we proposed. A revision of the well-known relation of Marshall is also proposed in order to determine the elastic modulus by means of one Knoop hardness test when the Vickers hardness is unknown.

Keywords: Indentation, Knoop, Vickers, ceramics

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1. Introduction

Conventional indentation tests have been widely used in determination of mechanical properties of materials, such us hardness, elastic modulus, tensile properties, toughness of bulk materials, and adhesion of ceramic coatings. The indenter can have various shapes such as spherical, conical or pyramidal. Among the most conventional indenters used are Vickers and Knoop pyramidal indenters that lead to the Vickers hardness *HV* and Knoop hardness *HK* respectively.

In conventional hardness testing, hardness of materials is defined as the ratio between the applied load *P* and a representative contact area between the indenter and the material. The representative contact area depends on the shape of the indenter and the definition of the representative area used. For Vickers hardness test, the indenter has a pyramidal with square base shape with a semi-apex angle θ between two opposite faces equal to 68° and the corresponding hardness number *HV* is calculated function of the true area A_T of the residual indent as follows :

$$\mathrm{HV} = \frac{\mathrm{P}}{\mathrm{A_T}} = \frac{2 \cdot \mathrm{P} \cdot \sin\theta}{\mathrm{d}^2} = 1.8544 \cdot \frac{\mathrm{P}}{\mathrm{d}^2} \qquad (1)$$

Where *d* is the average size of the measured diagonals of the indentation impression in mm and *P* is the applied load in *N*.

While Knoop hardness test use a lozenge-based pyramid with two different semi-apex angles θ_1=86°15, θ_2=65° along the length *L* and the width *b*, respectively. Knoop hardness number *HK* considers the projected area A_P of the residual indent. The projected area A_P is determined using the measured length of the indent by knowing the theoretical relationship between the length and the width of the impression as follows:

$$HK = \frac{P}{A_P} = \frac{2 \cdot P}{L^2} \cdot \frac{\tan\theta_1}{\tan\theta_2} = 14.229 \cdot \frac{P}{L^2} \quad (2)$$

where *L* is the measured long diagonal of the residual impression in mm and *P* the applied load in *N*.

Actually even if the hardness of a material is an intrinsic property, Knoop hardness and Vickers hardness test do not lead to the same hardness numbers. Many studies [1]–[5] reported that for the same material, Knoop hardness HK is usually smaller than Vickers hardness HV. Some authors compared the obtained hardness numbers for different materials and found that the ratio HV/HK varies between 1.05 and 1.15 for hard materials [1]. This difference has been subject to some interesting discussion [1], [5]–[8] and has been attributed to the unusual elastic recovery of Knoop impression [9] which is more pronounced for hard materials. Therefore, some authors suggested relationships between these two hardness numbers [1], [5]. However, hardness is an intrinsic property of materials and supposed to have the same value whatever the conventional hardness test used.

Thus the aim of this work is disambiguate hardness determination using conventional Vickers and Knoop hardness tests and shows how data can be used to obtain the same Vickers and Knoop hardness numbers representing an intrinsic property of materials.

2. Experimental

In order to analyze a high range of hardness, different hard materials covering a wide range of mechanical properties were studied in this work. Dense ceramics and glasses samples including one ultra-pure α-alumina Al_2O_3 (99.9% Almatis, Germany), silicon nitride Si_3N_4 (industrial origin), β-tricalcium phosphate (*β-TCP*) [10], oxinitride glass (G1N4 in Ref.[11]) and fused silica (industrial origin) were used. The ceramic specimens were mechanically ground with hard composite discs and diamond grinding suspension, while the glasses samples were dry ground using 1200 grit *SiC* paper in order to obtain fine ground plane surfaces. Then all specimens were carefully polished on resilient cloths with finer diamond pastes to produce an optical finish. Hardness tests were conducted using a commercial hardness tester (Zwick - Deutschland) equipped with Vickers and Knoop diamond indenters. Knoop and Vickers hardness measurements were conducted at test load ranging from 2 N to 20 N and more than 30 indentations were conducted on each specimen material using each indenter.
The dimensions of residual Vickers and Knoop impressions were measured by means of SEM type Hitachi S3500N in order to minimize optical measurement errors.

3. Results and discussion

Vickers hardness *HV* and Knoop hardness *HK* of each specimen material were calculated with Eq. (1) and Eq. (2) respectively. Obtained results were verified to be load independent which means that no indentation size effect ISE was observed since the applied loads are sufficiently high to avoid ISE effect [12], [13]. Obtained results are summarized in Fig. 1 in order to compare *HV* and *HK* numbers.
As several authors compared Vickers and Knoop hardness of different materials, we added in Fig. 1 available data in literature in order to complete the series of tests presented in this study.
Therefore, in the present work, we used, data provided by Ullner *et al.* [3], [4] who have tested several typical commercial ceramics, and the results reported by Mukhopadhyay et al. [2] on several based silicon nitride and SiAlON ceramics. Additionally, data on zirconium cristal were extracted from the published results of Gogotsi *et al.* [14]. Only results made on (001) plane of samples noted K-2 and K-3 in Ref. [14] were used in this study since complete information were provided only for these samples and do not exhibit ISE effect.
All data extracted from literature are summarized in table 1.

Table 1. Data related to the hardness numbers *HV* and *HK* for some ceramic materials reported in literature [2]–[4], [14].

Materials/Material code	HK (GPa)	HV (GPa)	E (GPa)	P (N)	L (µm)	b (µm)	Ref
	8.50	9.10	191	10	409.15	48.74	[2]
	11.30	12.00	243	10	354.85	42.00	[2]
ZrO_2 / K3	10.80	12.10	350	10	362.97	45.38	[14]
ZrO_2 / K2	11.20	12.20	350	10	356.43	44.51	[14]
	11.00	12.30	207	10	359.66	40.94	[2]
	11.60	12.70	241	10	350.23	40.93	[2]
	12.30	13.00	209	10	340.12	38.29	[2]
	11.30	13.10	247	10	354.85	41.41	[2]
	12.20	13.30	251	10	341.51	39.86	[2]
	12.30	13.80	269	10	340.12	39.96	[2]
	12.50	14.40	236	10	337.39	38.16	[2]
	12.80	14.50	223	10	333.41	37.11	[2]
	13.80	15.70	227	10	321.11	35.15	[2]
Al_2O_3 HPSN / A	14.30	15.80	410	19.62	441.84	54.45	[3]
SiAlON / F	14.10	16.00	290	19.62	444.97	51.50	[3]
Al_2O_3 VITOX / M	15.70	18.00	400	19.62	421.68	50.74	[3], [4]
Al_2O_3 RK / I	16.10	18.90	380	19.62	416.41	49.22	[4]
Al_2O_3 VITOX / K	17.10	19.90	310	19.62	404.05	45.13	[3], [4]
Al_2O_3 IKTS / J	17.40	20.70	380	19.62	400.55	46.49	[3], [4]
SiC IKTS / V	21.60	25.10	400	19.62	359.51	40.39	[3]

The figure 1 represents all the Vickers hardness data *HV* as a function the Knoop hardness data *HK* and shows without ambiguity that *HK* values are lower than *HV* values for all materials in the studied range of hardness. This was reported by several authors [1], [5], [15], [16] and attributed to the elastic deformations occurring during the indentation unloading.

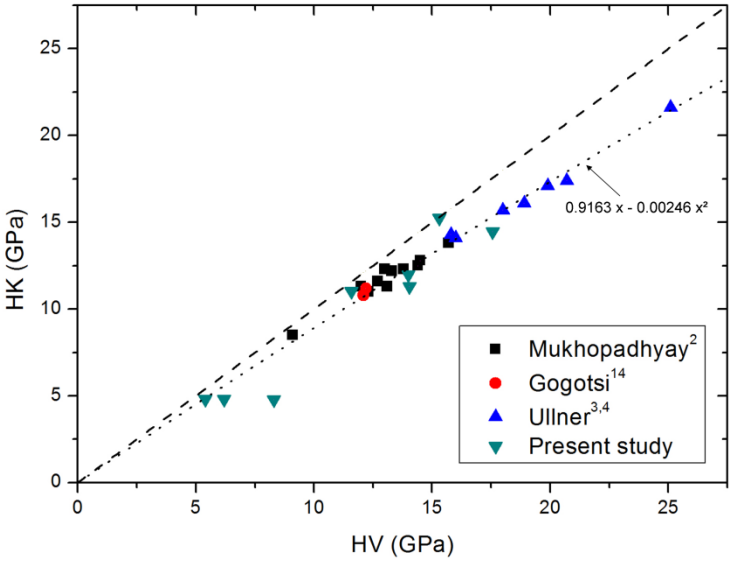


Fig. 1. Comparison between HV and HK.

In fact, during Vickers indentation, the indent diagonals remain approximately unchanged after load removal, and the elastic recovery occurs only along the indentation depth [17]. Whereas, Marshall *et al.* [9] reported that, after indenter withdrawal during Knoop indentation, a radial displacement of the impression also occurs (Fig. 2). The authors reported that, the elastic recovery occurs along the short axis and it is relatively important for materials with high ratio between hardness and elastic modulus *H/E*. They proposed a relationship between the recovered indentation size of the short diagonal and the ratio between the Vickers hardness *HV* and the elastic modulus *E* of the material.

$$\frac{b'}{L'} = \frac{b}{L} - \alpha \cdot \frac{H_V}{E} \qquad (3)$$

Where α is an empirical coefficient equals 0.45. The ratio of the short diagonal b to the long diagonal L at full load is given by the geometry of Knoop indenter and equal to 1/7.11. L' and b' are the long and the short diagonals of the residual impression after removal of load.

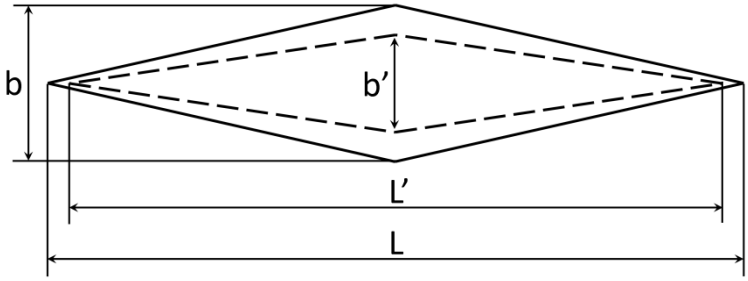


Fig. 2. Geometry of Knoop impression at full loading (straight lines) and after unloading (dashed lines).

Marshall *et al.* [9] found that the elastic recovery along the long diagonal is neglected compared to the elastic recovery along the short diagonal. Therefore they assumed that $L \approx L'$, which gives:

$$\frac{b'}{L} = \frac{1}{7.11} - 0.45 \cdot \frac{H_V}{E} \qquad (4)$$

It is noticeable from Eq. (4) that materials with high ratio *H/E* (e.g. glasses and ceramics) exhibit an important elastic recovery. Whereas, the elastic recovery can be negligible for materials with low ratio *H/E*. This explains why several authors [6]–[8] found that for ductile materials *HV* and *HK* are approximately the same and more the material is harder more the difference is important.

On the other hand, hardness is a contact area related parameter. Thus, it can have two different definitions since the representative contact area can be considered as the actual contact area at the maximum load, or as the residual area which represents the plastic impression. In the first case, hardness can be treated as the average contact pressure over the contact area. While in the second case, as it was defined by Tabor [18], it can be defined as resistance to plastic deformation by indentation. Anyway, there is a need to define a conventional hardness expression since in depth sensing indentation the second definition of hardness is commonly used [19].

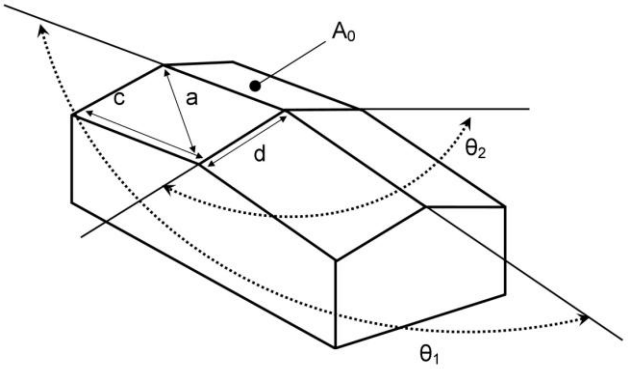


Fig. 3. Geometry of Knoop indenter.

Therefore, Chicot *et al.* [5] attribute the difference between hardness numbers *HK* and *HV* to the fact that Vickers hardness *HV* and Knoop hardness *HK* do not have the same definition of the representative area of impression used. Vickers hardness number uses the true indent area A_T while Knoop hardness number uses the

projected indent area A_P. Chicot *et al.* [5] suggested to use the same definition of the representative area used in the determination of hardness in order to compare the same physical quantities. The authors suggested using A_T in the calculation of both Vickers and Knoop hardness numbers. This means that Knoop hardness *HK* in Eq. (2) should be modified by taking into account A_T which could be deduced from the four sides of the indenter (Fig. 3). For this proposal one side of the indenter can be calculated by:

$$A_0 = \sqrt{s(s-a)(s-d)(s-c)}$$

$$\text{with } s = \frac{a+d+c}{2} \qquad (5)$$

a, *d*, *c* could be expressed as a function of *L* and of the apex angles θ_1 and θ_2 as following:

$$a = \frac{L}{2}\left(1 + \frac{\tan^2\theta_2}{\tan^2\theta_1}\right)^{1/2}, d = \frac{L}{2}\left(\frac{1}{\cos\theta_2 \tan\theta_1}\right)^{1/2}, c = \frac{L}{2\sin\theta 1} \qquad (6)$$

Therefore a new expression of *HK* can be obtained by considering the true area, A_T:

$$HK_T = \frac{P}{A_T} = \frac{P}{4 \cdot A_0} = 12.873\frac{P}{L^2} \qquad (7)$$

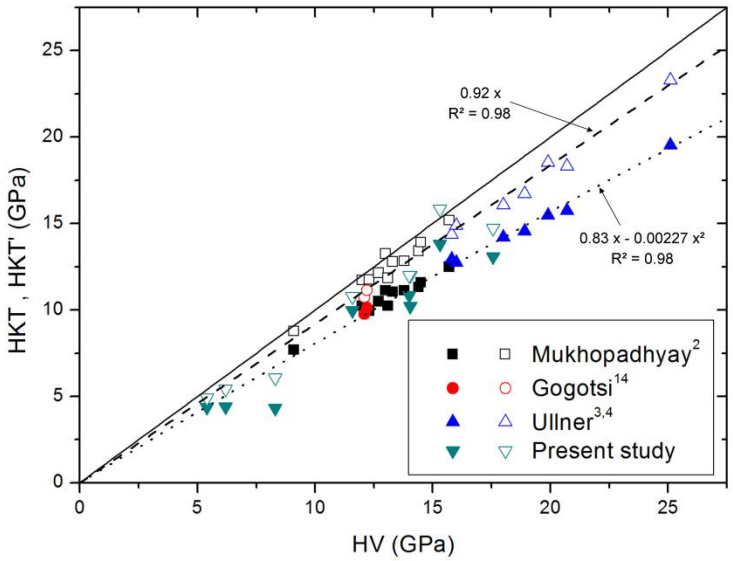


Fig. 4. Comparison between hardness numbers considering the true area of impression

Fig. 4 (colored dots) shows *HKT* values as a function of *HV*. It is clearly noticeable that there still exists a difference between the two numbers. Indeed *HKT* values are below the *HV* values over the entire studied range with an increase of the difference between for high hardness values. In fact, the assumption of Chicot *et al.* [5] is quite important in order to compare the same physical quantities; however they do not take into consideration the elastic recovery along the short diagonal during Knoop indentations which induces a variation of the apex angle θ_2 of the final impression. After elastic recovery the apex angle θ_2 become smaller by an amount depending on the ratio *H/E*, as it was reported by Marshall *et al.* [9] (Fig. 5) Therefore, we propose to compute the *HKT* basic on the new definition angle θ_2' which is related to the residual length of the short diagonal *b'*. Rearranging Eq. (3) assuming that there is no elastic recovery along the long diagonal such that *L*≈*L'*, gives:

$$\frac{b\prime}{b} = 1 - \alpha \cdot \frac{L}{b} \cdot \frac{HV}{E} \qquad (8)$$

Where *L/b*=7.11 and α=0.45 [9]. As it can be noticed from Fig. 5 the variation of the apex angle θ_2 can be deduced according to:

$$\frac{\tan\theta_2'}{\tan\theta_2} = \frac{b\prime}{b} \qquad (9)$$

Thus the residual apex angle θ_2' can be given by:

$$\tan\theta_2' = \frac{b'}{b} \cdot \tan\theta_2 \qquad (10)$$

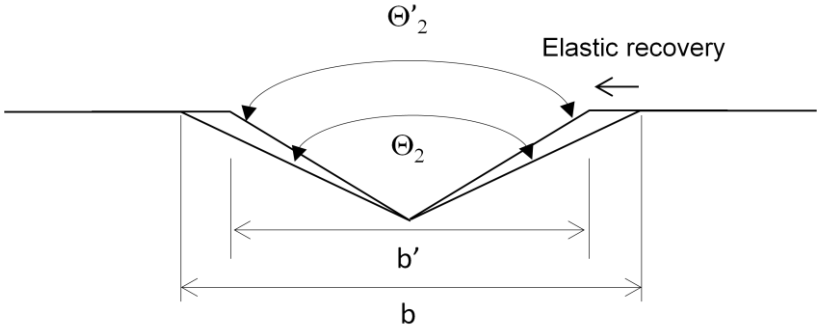


Fig. 5. Representation of the deformation near the short axis of Knoop impression at full load and after unloading.

Therefore the true area A_T' can be calculated basic on the residual apex angle θ_2' following Eq. (5) and Eq. (6) in order to obtain the Knoop hardness number *HKT'* considering the true plastic area A_T' instead of A_T in Eq. (7). The calculated *HKT'* values are compared to *HV* values in Fig. 4 (uncolored dots). Fig. 4 shows the influence of the correction brought to *HKT* by considering the radial elastic recovery. Indeed, as it can be seen from Fig. 4 a linear function is obtained by linear regression of *HKT'* function of *HV* with a strong correlation ($R^2 = 0.97$). Now, the two hardness numbers are almost equals even there exist a slight difference of 8% which is satisfactory. The slight difference can be attributed to the fact that the elastic recovery in depth (as it is neglected in Fig. 5.) also participate to the variation on the apex angle along the short axis θ_2, but also can induce a variation on the apex angle along the long diagonal θ_1 (Fig. 6). Unfortunately the elastic recovery in depth cannot be taken into account by conventional indentation with discrete application of loads but can be verified by means of instrumented indentation or 3D optical measurement which can give a drastic contribution in such test. Giannakopoulos and Zisis [20] have shown by numerical simulation that a slight elastic recovery can also occur along the long axis which can induce a variation on the apex angle θ_1.

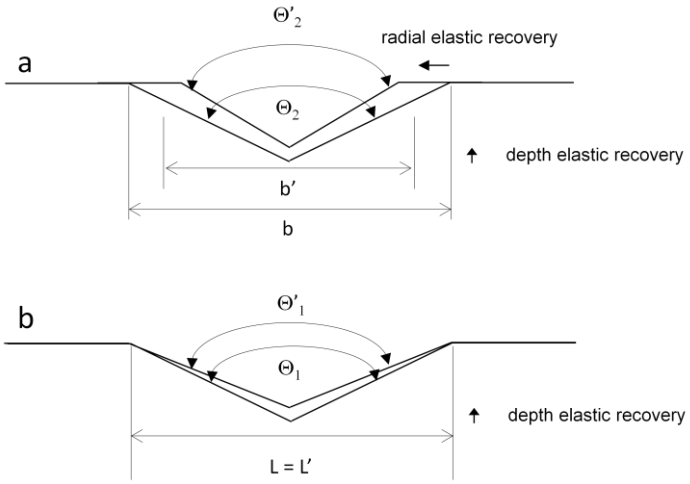


Fig. 6. Representation of the elastic recovery in depth and near the short axis of Knoop impression at full load and after unloading. (a) short diagonal ; (b) long diagonal.

Consequently, there exist another way to correlate between Vickers and Knoop hardness numbers obtained by discrete application of loads which can be also done by considering the projected area of the residual indent. This method has the advantage that it needn't knowledge of the true apex angles of the residual Vickers and Knoop impressions. Knowing that there is no elastic recovery along the diagonals of Vickers indent [17], Vickers Hardness number *HV* which used to be calculated using Eq. (1) should be changed to:

$$HV_P = \frac{P}{A_P} = \frac{P}{d^2/_2} \qquad (11)$$

where A_P is the projected residual area of the indent. This means that Vickers hardness values indicated in Fig. 1 could be recalculated by multiplying *HV* by 1.0785, which represents the ratio between the coefficients of Eq.

(11) and that corresponding to the standard Vickers hardness definition, i.e. 2 and 1.8544, respectively, in order to obtain HV_P.
In order to consider the elastic recovery occurring along the short diagonal in Knoop indentation, we propose to compute the Knoop hardness number considering the true projected area of the residual indent, which gives:

$$HK' = \frac{P}{A_P} = \frac{P}{L' \cdot b'/2} \qquad (12)$$

Fig. 7 presents the obtained *HK'* in comparison with HV_P. The obtained results were fitted with a linear regression and as it can be seen, it seems that both hardness numbers are approximately the same for a given material. This confirms the results obtained previously in considering the true area of contact residual impression, for which if Vickers hardness number *HV* has to be compared to Knoop hardness number *HK*, the same representative residual area must be considered in the calculation.

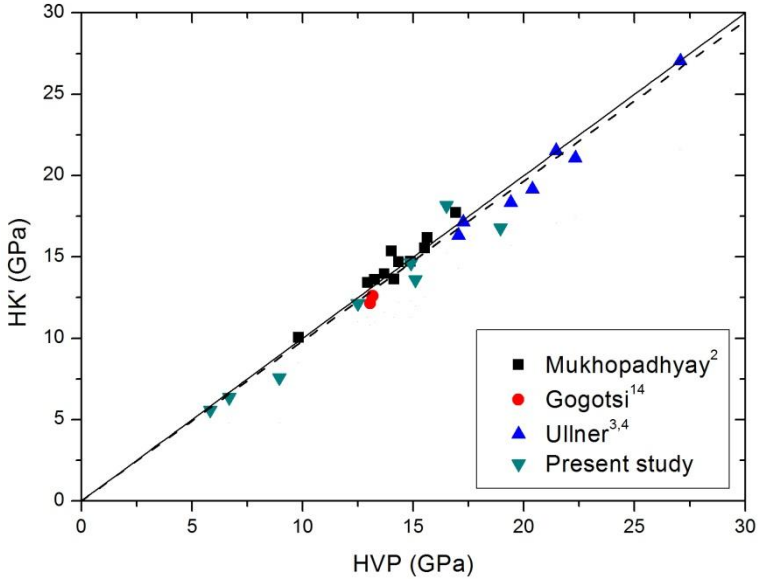


Fig. 7. Comparison between hardness numbers considering the projected plastic area.

Therefore, whatever the hardness test used, the same hardness number can be obtained as an intrinsic property of materials, if the true plastically deformed area is considered.
One of the main important purposes of these results is the simplification of elastic modulus evaluation by discrete application of loads. If this is simplest with depth sensing indentation, it is not the case where this technique is not available. The most commonly used method is the well-known Marshall's model (Eq. 4). The method consists in determining hardness using Vickers hardness test then evaluate the recovered indentation size of a Knoop indent. The results reported previously allow the simplification of this method by using one Knoop hardness test in order to obtain hardness and elastic modulus simultaneously. For this Eq. 4 can be changed by replacing *HV* by the HV_P which equals *HK'*:

$$\frac{b'}{L'} = \frac{1}{7.11} - \alpha \cdot \frac{HV_P/1.0785}{E} \qquad (13)$$

With $HV_P \approx HK'$ and $\alpha = 0.45$, then

$$\frac{b'}{L'} = \frac{1}{7.11} - 0.417 \cdot \frac{HK'}{E} \qquad (14)$$

The obtained *HK'* values allows us the calculation of elastic modulus *E* of the different studied materials with means of Eq. (14). The calculated elastic moduli are compared to the elastic modulus found in literature for the materials studied in this work and the values given by the references for data used from [2]–[4], [14].

Fig. 8 represents the efficiency of Eq. (14) by comparing the nominal elastic moduli to the values obtained by the new method of Eq. (14). It shows clearly that, the method proposed in this work can give reliable results of hardness and elastic modulus simultaneously by means of conventional Knoop hardness test.

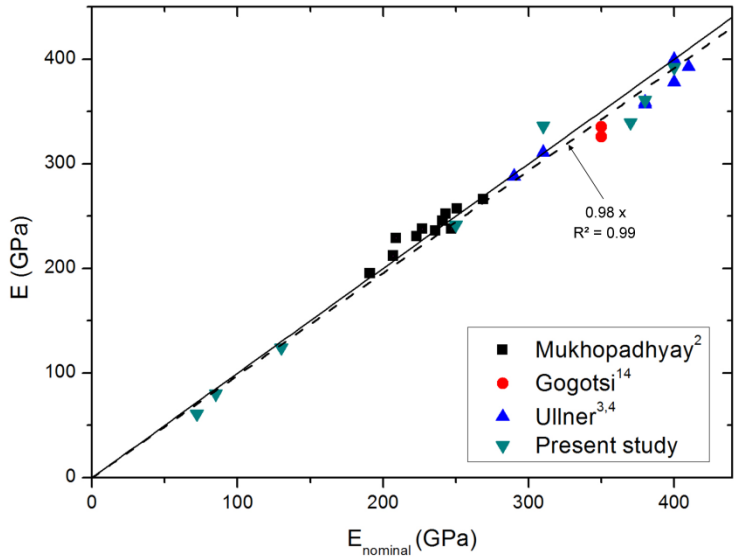


Fig. 8. Comparison between the nominal elastic moduli and the elastic moduli calculated with the new relation.

4. Conclusion

The discrepancy between Knoop and Vickers hardness numbers has been investigated. The difference between the conventional hardness numbers can be attributed to elastic recovery occurring in Knoop indent and to the difference of the definition of each hardness number. The Knoop and Vickers hardness numbers have been expressed on the same basis by taking into account the residual projected area which allows the obtaining of the same hardness number whatever the hardness test used. The main important conclusion of this paper is to confirm that hardness is an intrinsic property of materials independently of the hardness test employed. A revision of the Marshall's relation is proposed in order to determine the elastic modulus by using only Knoop hardness test instead of both Vickers and Knoop hardness tests.

As a secondary conclusion, Knoop hardness test can be a favorable candidate for mechanical characterization of brittle materials where Vickers hardness test is preferably avoided.

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