UDC 620.178.1:539.531: 539.533

DETERMINATION OF NANOHARDNESS AT A FIXED SIZE OF HARDNESS INDENT FOR THE ELIMINATION OF THE SIZE FACTOR

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To eliminate the indentation size effect (ISE), we propose to compare the nanohardness of different materials or a material in different structural states at equal sizes of hardness indents, characterized by a certain fixed displacement of the indenter h_f, rather than under a fixed load $P = const$, or recalculate the nanohardness for this fixed size. For the determination of the nanohardnesss H_f at h_f, the formula $H_f = H\left(\frac{h_f}{h}\right)^{m-2}$ is proposed, where m is the constant in the Meyer relation $P \sim h^m$. This approach enables us to compare more correctly the values of the hardness of different materials obtained under different loads.In the present work, 21 materials have been investigated, parameters that characterize the ISE have been determined and calculated, and the nanohardness has been calculated at a fixed displacement of the indenter h_f.

INTRODUCTION

Modern standard methods of mechanical (tensile, compression, and bending) tests do not always enable one to characterize the mechanical properties of new high-strength materials because of the small size of specimens and their insufficient plasticity at room temperature.

For the determination and characterization of the mechanical properties of brittle and low-plasticity materials, indentation methods are being extensively used. Indentation is the most commonly used method of studying the mechanical properties of coatings. However, it should be noted that the comparison of data obtained by these methods is not always correct in view of using different loads on the indenter and the existence of the size dependence of the hardness on the indent size (indentation size effect (ISE).

In recent years, instrumented indentation (with recording a load-indenter displacement curve) has been extensively used, which has made it possible to study the mechanical properties of materials in nanovolumes. This method revealed that the hardness increases with decreasing load on an indenter, which shows up most pronouncedly under small loads. The ISE simultaneously manifests itself as an increase in the nanohardness and a decrease in the plasticity characteristic (determined in indentation) with decreasing size of the nanohardness indent [1–15].

In contrast to standard mechanical tests (e.g., in tension, when the fracture of specimens of some materials occurs), in indentation, macroscopic fracture of a specimen does not occur, and, as a consequence, fracture does not influence the size effect in indentation. For these reasons, the physical nature of the size effect of hardness shows up better in nanoindentation under "more pure" conditions than those in the case of mechanical tests. In standard mechanical tests, it is possible to avoid the influence of the size factor on the mechanical properties by testing specimens of the same size for comparison of different materials or a material in different structural states. From this viewpoint, the determination of the hardness H should be performed at equal diagonals or equal depths of hardness indents h [16]. However, actually, in hardness measurements, a load on the indenter P is set, whereas the size of the indent and hardness are not only determined by the properties of the material, but also depend on the size factor.

This is why it is reasonable to standardize measurements of nanohardness in the sense that it must be determined at a certain fixed size of an indent or recalculated for this fixed size.

The authors consider the possibility of obviating the influence of the size factor on the value of the nanohardness by recalculation it for the standard (fixed in depth or in diagonal) indent size.

MATERIALS AND EXPERIMENTAL TECHNIQUE

In the present work, a large range of materials with different atomic structure and character of interatomic bonds has been investigated (Table). These are predominantly perfect single crystals or polycrystalline high-purity materials. Instrumented indentation was performed with the use of a Nano Indenter II (MTS Systems, USA) with a diamond indenter in the form of a Berkovich trihedral pyramid. The nanohardness and Young's modulus were calculated by the Oliver and Pharr methods [17]. In the most cases, the values of Young's modulus coincided with the tabular values.

Values of the indenter displacement h and nanohardness H at a maximum load on the indenter P, constants m (see (7)), n (see (8a)), i (see (10a)), and the value of the nanohardness H_f recalculated for a fixed displacement of the indenter h_f according to equation (12). Plasticity characteristic δ_H is given for the real value of H and for H_f (at h_f = 100 nm for ceramics and h_f = 1000 nm for metals)

Material	P_{max}, mN	E, GPa	h_{max}, nm	H, GPa	m	n	i	H_f, GPa, at h_f = 100 nm	H_f, GPa, at h_f = 1000 nm	δ_H, by H	δ_H, by H_f	h_f, nm
BeO*	10	400	181.5	12.8	1.58	-0.27	-0.42	16.5	6.2	0.77	0.70	h_f = 100 nm
TiN°	50	440	394.3	24.6	1.72	-0.16	-0.28	36.2	18.9	0.60	0.41	
Si_3N_4**	50	324	415.3	24.3	1.67	-0.20	-0.33	39.0	18.2	0.54	0.26	
NbC*	50	550	404.8	25.2	1.82	-0.10	-0.18	32.5	21.4	0.64	0.54	
NbC*	50	550	359.3	31.3	1.65	-0.21	-0.35	48.9	21.9	0.55	0.31	
ZrN*	50	400	400.7	24.3	1.65	-0.21	-0.35	39.7	17.6	0.57	0.31	
TiB_2**	50	540	308.2	44.1	1.63	-0.22	-0.37	66.7	28.6	0.42	0.16	
WC*	50	700	310.6	39.8	1.59	-0.26	-0.41	63.6	24.5	0.62	0.35	
LaB_6*	50	439	336.6	38.7	1.53	-0.30	-0.46	68.0	23.3	0.34	0.04	
β -SiC*	50	460	323.2	44.3	1.70	-0.17	-0.30	62.8	31.6	0.29	0.09	
ZrC**	50	480	386.0	26.4	1.63	-0.22	-0.37	43.3	18.6	0.57	0.31	
B_4C°	10	500	123.3	48.9	1.64	-0.22	-0.36	52.8	22.8	0.23	0.19	
Al_2O_3*	10	409	144.9	33.3	1.64	-0.22	-0.36	38.0	16.6	0.41	0.33	
MgO*	50	310	584.0	9.46	1.74	-0.15	-0.26	15.1	8.2	0.76	0.79	h_f = 1000 nm
W*	10	420	301.3	6.10	1.85	-0.08	-0.15	7.2	5.1	0.91	0.93	
Mo*	50	324	931.2	3.21	1.71	-0.17	-0.29	6.1	3.1	0.94	0.95	
Cr*	50	279	1025.3	2.63	1.66	-0.20	-0.34	5.7	2.6	0.95	0.95	
Nb**	50	104	1460.2	1.26	1.84	-0.08	-0.16	1.9	1.3	0.96	0.96	
Ta**	50	185	1259.2	1.74	1.75	-0.14	-0.24	3.2	1.8	0.96	0.96	
Cu*(111)	62.5	170	2100.8	0.66	1.72	-0.16	-0.28	1.6	0.8	0.98	0.97	
Al**	120	70	3148.0	0.66	1.73	-0.16	-0.27	1.7	0.9	0.96	0.95	

* – single crystal, ** – polycrystalline, ° – individual grain

PHENOMENOLOGICAL DESCRIPTION OF THE SIZE DEPENDENCE OF HARDNESS

In [10, 11, 18, 19], the plastic deformation of a material in indentation is described by the Brown rotational model, and, in this case, plastic flow is considered as a displacement along semicircular slip surfaces with a center at the tip of the indenter. In the case of this mechanism of deformation, geometrically necessary dislocations (GNDs), which lead to strain hardening, are initiated. In this model, the ISE is explained by an increase in the density of dislocations as a result of a decrease in the indent size and by the motion of dislocations around a smaller slip circle. An expression obtained in this model for a Vickers indenter has the form:

$$H = A^* \alpha^* Gb \left(\rho_0 + \frac{ctg\gamma}{bd} \right)^{1/2}, \qquad (1)$$

where H is the Meyer hardness; A^* is the ratio of the hardness (normal stress under the indenter) to the yield strength; for metals, $A^* \approx 3$ and $\alpha^* \approx 1/3$; G is the shear modulus; b is the Burgers vector of a dislocation, ρ_0 is the initial dislocation density, d is a diagonal of the indent, and γ is the angle between a face and the axis of the pyramidal indenter.

In the frequently used best-known Nix–Gao model [4], it is assumed that indentation is accompanied by the formation of dislocation loops of geometrically necessary dislocations with a Burgers vector b, normal to the surface of the specimen. With regard for both the geometrically necessary and statistically distributed dislocations, the following expression was obtained:

$$\frac{H}{H_0} = \sqrt{1 + \frac{\rho_G}{\rho_S}} = \sqrt{1 + \frac{h_0}{h}}, \qquad (2)$$

where H_0 is the hardness in the absence of geometrically necessary dislocations, ρ_G is the density of geometrically necessary dislocations, ρ_S is the density of statistically distributed dislocations, and h_0 is the size parameter of the dependence of ρ_S in terms of H_0.

Taking into account that $d \sim h$, we can rewrite equations (1) and (2) in the form:

$$H^2 = C\left(1 + \frac{C_1}{h}\right), \qquad (3)$$

where C and C_1 are constants.

The dependence of this type was substantiated in [4, 5], but, in many cases, the dependence $H^2 \sim \frac{1}{h}$ has a bilinear character, and the values of the constants C and C_1 can differ substantially from their theoretical values.

It is likely that the presented models are fairly successful, but only in a first approximation to the dislocation mechanism of deformation in indentation, the more so because mechanisms of deformation can

differ significantly for different crystalline and noncrystalline materials.

For this reason, it is difficult to use the above equations to recalculate the values of the hardness and nanohardness for one load on the indenter to their values for another load.

In the present work, we consider a more general phenomenological approach to the ISE, which does not require knowledge of the dislocation mechanism of deformation in indentation. In this approach, the empirical power dependence $P \sim h^m$ (where $m \approx const$), repeatedly substantiated in experiments [16, 20–23], is used, and the nature of the size dependence of the hardness is discussed in connection with the relation between the elastic ε_e and plastic ε_p strain of the material under an indenter is discussed.

It is known that at indentation of materials by conical and pyramidal indenters, the total strain is described by the following equation:

$$\varepsilon_t = \varepsilon_e + \varepsilon_p \approx const\ . \tag{4}$$

In what follows, we discuss the mean values of the strain on the indenter – specimen contact area in the direction of action of the load. In indentation, the Hooke law holds in the form [24]:

$$\varepsilon_e = (1 - \nu - 2\nu^2)\frac{H}{E}, \tag{5}$$

where H is the Meyer hardness,

$$H = \frac{P}{S} = \frac{\alpha P}{h^2}, \tag{6}$$

P is the load on the indenter, S is the area of the projection of the hardness indent, E – is Young's modulus, ν is Poisson's ratio, h is the penetration depth of the indenter, and α is the coefficient of the indenter form.

With decrease in the size of the hardness indent (or with decrease in the load P), the plastic deformation ε_p is hindered and decreases. The suppression of plastic deformation is caused by an increase in the dislocation density, difficulties in operation of dislocation sources, and a decrease in the mean path length of dislocations. At the same time, the elastic strain ε_e is determined by the Hooke law (5) independently of the indent size. In the case of pyramidal indenters, $\varepsilon_t \approx const$ and is determined by the angle γ at the tip of the indenter between the axis of the pyramid and its face. This is why the suppression of plastic deformation and the decrease in the value of ε_p in accord with (4) leads to an increase in ε_e and, hence, according to (5), to an increase in the hardness [14, 25].

Let us represent the relation $P \sim h^m$ in the form:

$$P = K\left(\frac{h}{h_0}\right)^m, \tag{7}$$

where K is a constant, h_0 is a displacement equal to 1 in the used system of units. For nanohardness, it is reasonable to take h_0 = 1 nm. The value of the exponent m is usually below 2 [16]. In [20–22], it was shown that the parameter m decreases linearly with increasing ratio H/E, where E is Young's modulus. Since with decrease in the load on the indenter, the nanohardness increases substantially, this can be one of the reasons of an insignificant reduction in m with decreasing load P.

Using expressions (6) and (7), we obtain:

$$H = K_1 \cdot P^n, \tag{8}$$

where $n = 1 - 2/m$ (8a)

($n < 0$, i.e., H decreases with increasing load P), and

$$K_1 = \frac{\alpha \cdot K^{2/m}}{h_0^2} \approx const\ .$$

It is important to note that relation (8) usually holds fairly well for single crystals. For polycrystals, this expression can be used if the size of the hardness indent is smaller than the grain size [23]. At a smaller grain size, the length of the slip plane is determined by the grain size D, and the size dependence of the hardness must also take into account D [26]. In nanohardness measurements, the relation $h << D$ holds for most materials, which enables us to use relation (8) to describe the size dependence of the nanohardness.

Along with dependences (1), (2), and (3), in the literature, the ISE is commonly characterized by the power dependence $H=f(h)$ [5, 23, 27]:

$$H = Ah^i, \tag{9}$$

where A and i are constants.

In view of (6) and (7), we get:

$$A = \frac{\alpha \cdot K}{h_0^m} \tag{10}$$

and

$$i = \mathrm{m} - 2\ . \tag{10a}$$

According to [27], the experimental values of i for different materials range from –0.12 to –0.32.

As an example, the authors investigated the change in the nanohardness H depending on the load P for single crystal Cu (111) (Fig. 1) earlier.

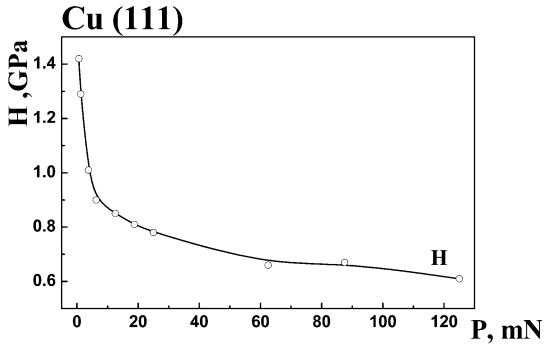

Fig. 1. Influence of the load on the indenter P on the nanohardness H of single crystal Cu (111) [25]

It is seen in Fig. 1 that a decrease in the load P leads to a substantial increase in H. This phenomenon shows up particularly noticeably under $P < 6$ mN ($h < 500$ nm).

The sharp dependence of the nanohardness on the load P and depth of penetration of the indenter h raises the problem of the necessity of having a technique for recalculating the value of the nanohardness for one load to its value for another load for practical application. The standardization of measuring the nanohardness in

the sense that it must be determined at a certain fixed size of an indent or recalculated for this fixed size is also reasonable.

For the recalculation of the value of the nanohardness for one load P_1 to its value for another load P_2, we can use the following relation obtained for the microhardness in [16]:

$$H_2 = H_1\left(\frac{P_2}{P_1}\right)^n \qquad (11)$$

(This relation is easily obtained from expression (8) presented in the present work.)

The constant m can be determined by the formula $m = \frac{d\log P}{d\log h}$ if the hardness was determined under different loads P. The constant m can also be approximately determined from a curve of loading in the P–h coordinates if this curve is represented in the form $P = K^* \cdot h^{m*}$ and is recorded at a sufficiently large maximum load P.

To determine the nanohardness H_f at a fixed size of an indent of h_f, we can use relation (9), from which we get:

$$H_f = H\left(\frac{h_f}{h}\right)^i. \qquad (12)$$

Therefore, by the value of H_f, we can calculate the size dependence of the hardness by the following equation:

$$H = H_f\left(\frac{h}{h_f}\right)^i \qquad (12a)$$

Here, H is the hardness at a certain displacement of the indenter h. It is naturally desirable that h differs not significantly from h_f because the value of m depends, while insignificantly, on h. In nanohardness measurement, the choice of h_f that can be used for all materials is complicated by the large difference in hardness between materials of different types.

This approach to the determination of the nanohardness at a fixed size of an indent, rather than under a fixed load P, makes it possible to eliminate the influence of the size factor on the nanohardness, i.e., it enables us to compare more correctly the hardness of materials.

EXPERIMENTAL VALUES OF CONSTANTS THAT CHARACTERIZE THE ISE

In the present work, using the obtained results, we calculated the constants m, n, and i, which characterize the ISE, and the nanohardness at a fixed displacement of the indenter h_f. Plasticity characteristic δ_H [14, 15, 24] was calculated as well. The obtained results are presented in Table.

On the basis of the obtained experimental data, a plot of the dependence of the constant m on the ratio of the hardness H to Young's modulus E was constructed (Fig. 2).

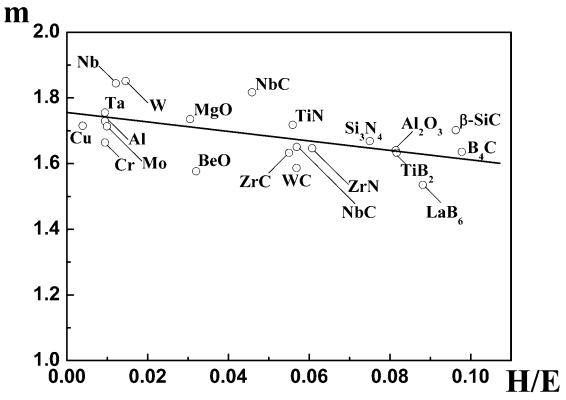

Fig. 2. Dependence of the constant m on the ratio of the hardness to Young's modulus H/E

As has been noted, in microhardness tests, m decreases linearly as the ratio H/E increases. In contrast to microhardness tests, in nanoindentation, a large deviation of the values of m from a linear dependence is observed, which can be due to the essential value of the ISE in nanoindentation.

In [28], data on the dependence of m on H/E for macrohardness were generalized. It was shown that a decrease in m is observed, but the value of m is somewhat higher than those for microhardness and nanohardness and practically attains 2.

In the present work, the decrease in the parameter m with increasing ratio H/E is also substantiated and is beyond doubt. In this case, the constant n and the absolute value of the constant i increase, and, hence, a stronger significant influence of the size factor on low-ductile and hard materials is observed.

The results of calculation of H_f show that the ISE is fairly substantial in nanohardness measurements. It is seen that, as was expected earlier, a wide spectrum of investigated materials cannot be correctly recalculated at single h_f. The recalculated values of the hardness of brittle and low-plasticity materials correlate most acceptably with the corresponding measured values at h_f = 100 nm. At the same time, for plastic materials with a low hardness the value h_f = 1000 nm is preferred.

Note that the recalculated values enable us to compare the hardness of a wide range of materials in the absence of the influence of the size factor.

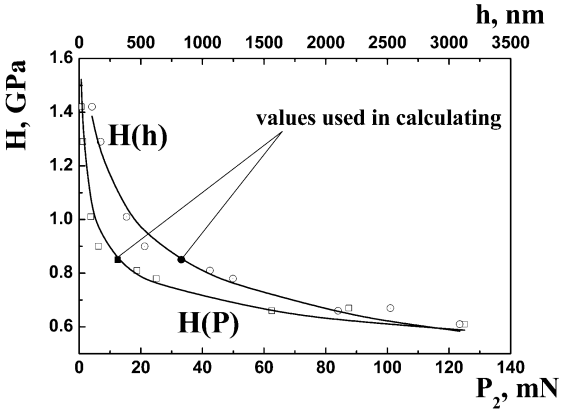

Fig. 3. Curves of the dependence of the nanohardness H on the load on the indenter P and the displacement of the indenter h for single crystal copper (111) and experimental dots obtained in [29]

To check the developed notions, the authors present dependences *H(P)* (calculated by equation (11) at H_1 = 0.85 GPa, P_1 = 12.5 mN, and n = –0.16) and *H(h)* (calculated by equation (12a) at H_f = 0.85 GPa, h_f = 827 nm, and i = –0.28) for single crystal copper. The obtained results are shown in Fig 3.

It is seen that the curves constructed on the basis of the calculated values of the hardness coincide satisfactorily with experimental results obtained in [29].

One can see from the table 1, that the ISE is not very essential for the plasticity characteristic δ_H of metals, but for ceramics the influence of the ISE on the δ_H is very strong. This problem requires further study and discussion.

CONCLUSIONS

1. Fracture does not influence on the size effect in indentation in contrast to standard mechanical tests (e.g., in tension, when, in some materials, the fracture of a tested specimens occurs). For these reasons, the physical nature of the size effect shows up in nanoindentation under "more pure" condition, than those in uniaxial mechanical tests. The size effect in nanoindentation (an increase in the hardness with decreasing load on the indenter) can be explained by the fact that plastic deformation is inhibited with decrease in the size of the indent, but the elastic strain is independent on the indent size at condition that the total strain is nearly constant $\varepsilon_t \approx const$ and is determined by the shape of the indenter.

2. The phenomenological approach to the ISE, in which the power dependence of the load on the indenter P on the displacement of the indenter h in the form of the Meyer relation $P = const \cdot h^m$, (where m is a constant) is used, enables us to describe the ISE for single crystals by the equations $H = K_1 \cdot P^n$ and $H = Ah^i$, where $n = 1 - 2/m$ and $i = m - 2$. As a rule, for polycrystals, these relations hold if the size of the indent is several times smaller than the grain size, which usually takes place in determination of nanohardness.

3. The nanohardness H_1 obtained under a load P_1 can be recalculated to the nanohardness H_2 under a load P_2 by the relation $H_2 = H_1\left(\frac{P_2}{P_1}\right)^n$.

4. To eliminate the size effect, it is reasonable to perform a comparison of the nanohardness of different materials or a material in different structural states not under $P = const$, but at equal sizes of hardness indents, characterized by some fixed displacement of the indenter h_f.

The recalculation of the nanohardness H obtained at some value of h to the nanohardness H_f at a fixed size of the indent h_f can be performed by the expression $H_f = H\left(\frac{h_f}{h}\right)^i$. As h_f, it is proposed to use the value 1000 nm for metals and 100 nm for ceramics and other high-strength materials.

5. The performed investigations have shown that the formulas proposed by the authors describe fairly well the experimental data and can be used to eliminate the influence of the size factor on the hardness of materials. We believe that, in subsequent works, it is reasonable to perform correction of this type of measured results by recalculation at h_f for the correct comparison of the values of the hardness obtained in different works under different loads.

6. It was shown that influence of the ISE on the plasticity characteristic δ_H of ceramics is very strong. This problem requires further study.

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Article received 16.01.2015

ОПРЕДЕЛЕНИЕ НАНОТВЕРДОСТИ ПРИ ФИКСИРОВАННОМ РАЗМЕРЕ ОТПЕЧАТКА ТВЕРДОСТИ ДЛЯ УСТРАНЕНИЯ МАСШТАБНОГО ФАКТОРА

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Сравнение нанотвердости различных материалов или одного материала в различных структурных состояниях для устранения масштабной зависимости твердости ISE (Indentation Size Effect) предлагается проводить не при нагрузке $P = const$, а при одинаковом размере отпечатка твердости, характеризуемом некоторым фиксированным значением перемещения индентора $h_ф$, или пересчитывать на этот фиксированный размер. Для определения нанотвердости $H_ф$ при $h_ф$ предложена формула: $H_\phi = H\left(\frac{h_\phi}{h}\right)^{m-2}$, где m – константа в соотношении Мейера $P \sim h^m$. Такой подход позволяет более корректно сопоставлять величины твердости материалов, полученные при разных нагрузках. В работе исследован 21 материал, определены и рассчитаны параметры, характеризующие ISE, а также рассчитана нанотвердость при фиксированном значении перемещения индентора $h_ф$.

ВИЗНАЧЕННЯ НАНОТВЕРДОСТІ ПРИ ФІКСОВАНОМУ РОЗМІРІ ВІДБИТКА ТВЕРДОСТІ ДЛЯ УСУНЕННЯ МАСШТАБНОГО ФАКТОРУ

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Порівняння нанотвердості різних матеріалів або одного матеріалу в різних структурних станах для усунення ISE (Indentation Size Effect) пропонується проводити не при навантаженні $P = const$, а при однаковому розмірі відбитка твердості, який характеризується деяким фіксованим значенням переміщення індентора $h_ф$, або перераховувати на цей фіксований розмір. Для визначення нанотвердості $H_ф$ при $h_ф$ запропоновано формулу: $H_\phi = H\left(\frac{h_\phi}{h}\right)^{m-2}$, де m – константа в співвідношенні Мейера $P \sim h^m$. Такий підхід дозволяє більш коректно порівнювати величини твердості матеріалів, отримані при різних навантаженнях. У роботі досліджено 21 матеріал, визначені і розраховані параметри, що характеризують ISE, а також розрахована нанотвердость при фіксованому значенні переміщення індентора $h_ф$.