

Reducing the influence of the surface roughness on the hardness measurement using instrumented indentation test

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Abstract. The instrumented indentation method requires the sample surface to be flat and smooth; thus, hardness and elastic modulus values are affected by the roughness. A model that accounts for the isotropic surface roughness and can be used to correct the data in two limiting cases is proposed. Suggested approach requires the surface roughness parameters to be known.

1. Introduction

Over past several decades, instrumented indentation technique has widely been used for the materials mechanical properties measurement at the submicron and nanoscale length scale level [1]. According to this method, the hardness value is calculated as a relation of applied normal load F to the contact area A_c achieved during the corresponding indenter penetration into the sample surface. The contact area is not measured directly, but is defined by the so-called area function $A_c = f(h_c)$, where h_c is a contact depth estimated during experiment from the loading-unloading diagram. Typically, area function is calibrated on a reference sample using the series of indentations.

The method requires sample preparation, demanding that the surface has to be flat and smooth. In practice, this condition is considered to be met when the indentation depth is bigger than $20 \cdot R_a$, where R_a is the arithmetic average of the roughness profile. Such a criterion gives an uncertainty less than 5% [2]. Violating this rule leads to incorrect estimation of the contact area and, consequently, changes measured hardness value.

However, some samples, for example thin rough films and coatings, do require the indentation depth to be small, comparable to R_a and apparently smaller than $20 \cdot R_a$. Even apart from such examples, each sample roughness contributes to the measured values of hardness and elastic modulus, not only increasing the data scattering, but also biasing the mean values. Therefore, the proper model for the error estimation and correction should be useful.

2. Existing model and current approach

The idea to make an account for the roughness is to find the cases, where the interaction with rough surface can be reduced to the smooth contact but with corrected value of the contact area. A simple idea for the rough surface with relatively “frequent” peaks is suggested in [3]. The approach implies that the number of peaks being squeezed beneath the indenter tip is big enough and the mean surface slope is bigger than the indenters pyramid angle. The last assumption leads to the fact that the initial contact occurs roughly at $dz \sim 2.5 \cdot R_a$ above the mean surface line (for the normal-distributed heights). Indenter flattens the protruding asperities to the mean line and situation reduces to the flat



smooth surface contact with the only correction $h_c \rightarrow h_c - dz$. The contact area A_c is recalculated according to the calibrated contact area function.

New approach proposed in the current work extends the above idea and considers the surfaces with the range of average slopes that are not necessarily bigger than the apex angle of the indenting pyramid. Moreover, another limiting case of a shallow contact with the slope of the single roughness asperity is also considered. The last case reduces to the contact with smooth inclined surface.

To create such kind of model, one needs to describe the surface spatial frequencies. This can be done either by using a PSD (power spectral density) or an autocorrelation function. In the current approach, we assumed a Gaussian autocorrelation function. The correlation tensor for the 2D-surface was defined in the following way:

$$C_{x_i, y_i, x_j, y_j} \sim \left(\exp \left(-\frac{(x_i - x_j)^2 + (y_i - y_j)^2}{\sigma_{xy}^2} \right) + 10^{-3} \left(1 - \frac{|x_i - x_j|}{L_x} \right) \left(1 - \frac{|y_i - y_j|}{L_y} \right) \right)$$

Normalizing constant was chosen to be one unit; the overall 2D array was renormalized afterwards to have some certain value of standard deviation σ_z for the height distribution. The last term in this expression was added to make the tensor condition number small enough, so that the future numerical calculation is possible.

To generate a 2D surface we've used a tensor A package available for R software [4]. Cholevsky decomposition was used to find a tensor $L_{a,b,i,j}$ so that

$$L_{i,j,a,b} L_{k,m,a,b} = C_{i,j,k,m} \quad (1)$$

For the uncorrelated array of normal-distributed random numbers $Z0_{i,j}$, the following transformation gives normally distributed correlated array:

$$L_{i,j,a,b} \cdot Z0_{i,j} = Z_{a,b} \quad (2)$$

7500 different $Z_{a,b}$ arrays were generated. Each of them has 128x128 points, σ_{xy} parameter was set to be equal to 4. Heights of each of 7500 arrays were rescaled to have different ratios of σ_z/σ_{xy} . 26 values of σ_z/σ_{xy} in the range of [0.02, 6] were used.

3. Contact point bias correction

For each array, an approaching of Berkovich type three-sided pyramid till the initial contact was modeled. Wolfram Mathematica software was used. The coordinate dz of the initial contact from the mean surface plane was calculated. The probability density functions (PDFs) of normalized value of dz/σ_z are presented in the Figure 1.

As it follows from the figure for surfaces with the “small slopes” i.e. $\sigma_z/\sigma_{xy} = 0.02 \dots 0.2$, average value of dz is almost equal to zero, which means that depth measurements for such kind of surface are not biased. In this case, the error in hardness measurements more likely comes from the local slope of surface; this effect will be considered later.

As the ratio of σ_z/σ_{xy} grows, bias in the average value of $\langle dz \rangle/\sigma_z$ becomes evident. This corresponds to the situation, when the initial contact occurs between the surface and pyramid's face instead of the pyramid tip. To characterize this effect, the center of mass of each PDF was calculated. Corresponding dependence of $\langle dz \rangle/\sigma_z$ vs σ_z/σ_{xy} is presented in Figure 2.

Interpolation function $f(\sigma_z/\sigma_{xy})$ of $\langle dz \rangle/\sigma_z$ versus σ_z/σ_{xy} dependence can be used to correct the indentation data. The correction implies recalculation of data with the following change: $h_c \rightarrow h_c - \sigma_z \cdot f(\sigma_z/\sigma_{xy})$. As it follows from the Figure 2, in the case of $\sigma_z/\sigma_{xy} \gg 1$ the value of $\langle dz \rangle$ is shifted to $3\sigma_z$, i.e. corresponds to “three sigma rule” and to the case considered in work [3].

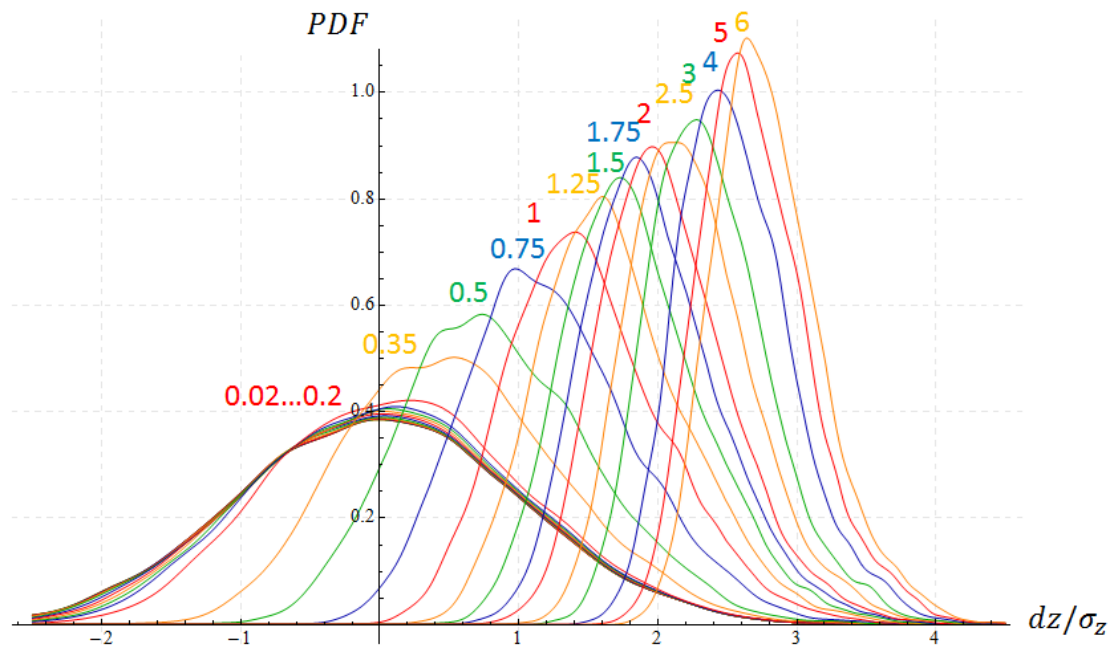


Figure 1. PDFs of normalized shift of the contact point dz/σ_z for the different values of σ_z/σ_{xy} .

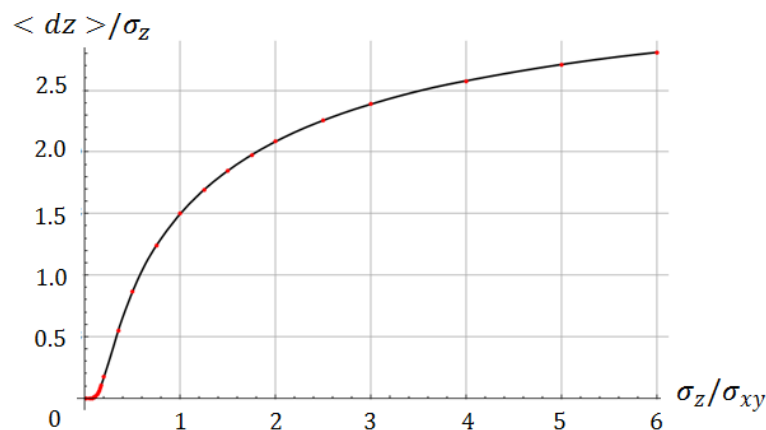


Figure 2. Average normalized contact point shift vs σ_z/σ_{xy} parameter.

4. Average slope correction

In case of small σ_z/σ_{xy} values (i.e. $\sigma_z/\sigma_{xy} \lesssim 0.1$) in average the initial contact occurs on the surface mean plane, so that the value $\langle dz \rangle$ is equal to zero, but still there is an error in hardness and elastic modulus measurements that comes from the local slope.

To make an account for the described effect, let us consider the Berkovich pyramid contacting the inclined surface. To fix the idea, let us suppose that the pyramid's height is aligned along the vertical direction and the surface is inclined so that its normal has the angles φ and θ in spherical coordinates: φ is calculated from the OX axis, the pyramid edge being aligned along OY axis; θ is calculated from the pyramid height, pyramid's tip looking down.

In this case, it can be shown that for the given depth projected area of contact A_p is related to the flat surface contact area (for the same depth) A_0 according to the following relation:

$$\frac{A_p}{A_0} = \frac{\cot[\alpha]^3}{(\cot[\alpha] + 2\sin[\varphi]\tan[\theta])(\cot[\alpha]^2 - 2\cot[\alpha]\sin[\varphi]\tan[\theta] - (1 + 2\cos[2\varphi])\tan[\theta]^2)} \quad (3)$$

where α is the angle between pyramid's height and face. As long as the surface is considered isotropic, it is necessary to average this function across all φ values:

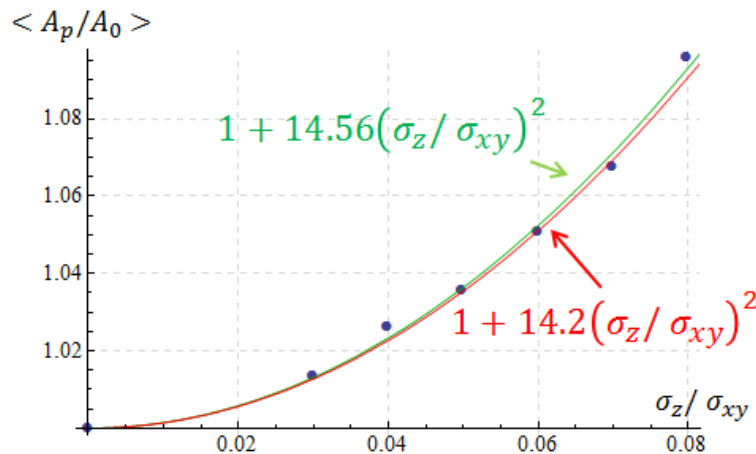


Figure 3. Correction factor for contact area in the case of single-asperity indentation: analytical expression (red line) and numerical experimental verification (green line).

$$\langle \frac{A_p}{A_0} \rangle = \frac{1}{2\pi} \int_0^{2\pi} \frac{A_p}{A_0}(\varphi, \theta) d\varphi \quad (4)$$

Up to the second-order terms, the integration gives:

$$\langle \frac{A_p}{A_0} \rangle \approx 1 + 3tg(\alpha)^2 \tan(\theta)^2 \quad (5)$$

For the Berkovich indenter $\alpha = 65.3^\circ$, which leads to the following relation:

$$\langle \frac{A_p}{A_0} \rangle \approx 1 + 14.2 \tan(\theta)^2 \quad (6)$$

To show the way to correct the data with the obtained equation, the following comparison with the numerical simulation is useful. We have used 300 numerically simulated surfaces for each of the 6 different values of σ_z/σ_{xy} in the range $0 < \sigma_z/\sigma_{xy} < 0.1$. Penetration of Berkovich type pyramid into each surface was modeled up to the depth of $\sigma_z/75$. The ratio of $\frac{A_p}{A_0}$ has been averaged across four depth values for each surface and then across all of the surfaces for the given σ_z/σ_{xy} value. The resulted points are presented in Figure 3 by dots. The experimental points were fitted with the parabola curve, which led to the following relation: $\langle A_p/A_0 \rangle = 1 + 14.56 (\sigma_z/\sigma_{xy})^2$.

The coincidence between two curve leads to the conclusion that the parameter σ_z/σ_{xy} is simply an equivalent of the $\tan(\theta)$ and the experimental data can be corrected by multiplying each of the calculated area value by the factor of $1 + 14.2 (\sigma_z/\sigma_{xy})^2$.

5. The use of the model for the experimental data

The model was tested on the several polycarbonate samples with different roughness. The rough surface was obtained using the sandpaper with the different grit parameters: 320, 500, 800, and 1200. Statistical parameters of the surface were obtained using AFM Ntegra Prima with installed both upper and lower piezo-scanners allowing the scanning area up to $200 \times 200 \mu\text{m}^2$. The σ_z values were determined from the RMS value, σ_{xy} values were determined from the width of autocorrelation function base. Some auto-correlation functions appeared to be quite antisymmetric, so all widths were averaged across two perpendicular directions.

A series of indentations was performed on each sample, load value and number of indentation are given in Table 1. NanoScan 4D instrumented indentation tester was used.

The hardness value without correction is calculated as an average value from all the indentations: $\langle H \rangle = \frac{1}{N} \sum_i H_i$. According to the model described above, corrected value of hardness was calculated as

follows: $H_{corr} = F/A_c(\frac{1}{N}\sum_i h_{ci} - \sigma_z \cdot f(\sigma_z/\sigma_{xy}))$, where $A_c(h_c)$ is the indenter area function. For the sample with grit parameter 320 and force 50 mN, the averaged value $\langle H \rangle = 0.15$ GPa was even closer to the smooth surface value of 0.21 GPa than the corrected value, which is 0.14 GPa. Such a difference can be explained by the fact that here we compared average hardness with hardness calculated from the average contact depth. This was done intentionally, because namely the average hardness is value that is reported for the substance. Calculation of non-corrected value from the mean contact depth $F/A_c(\frac{1}{N}\sum_i h_{ci})$ for this series would give the value of 0.13 GPa, which is farther from the “true” value of 0.21 GPa.

Table 1. Rough surface parameters and hardness measurements results.

Sample grit parameter	Amount of indents	σ_z , nm	σ_{xy} , nm	σ_z/σ_{xy}	F, mN	$\langle H \rangle$, GPa (non-corrected)	H_{corr} , GPa (corrected)
180	101	7382	27380	0.27	100	0.1	0.18
	99				1400	0.16	0.19
320	100	1770	9720	0.18	100	0.14	0.14
	100				1400	0.18	0.18
320	64	1250	8558	0.15	10	0.15	0.15
	43				50	0.15	0.14
	22				200	0.17	0.19
500	47	950	4327	0.22	10	0.16	0.2
	45				50	0.2	0.23
	12				200	0.19	0.21
800	100	1104	4380	0.25	100	0.14	0.15
	100				1400	0.16	0.17
1200	52	500	2806	0.18	10	0.2	0.21
	26				50	0.19	0.2
	16				200	0.19	0.2

6. Conclusion

Roughness biased value of hardness can be corrected using an information about the surface topography, namely the parameters that describe surface auto correlation function and height distribution. Based on these parameters, the proposed model can be used to correct the measured hardness value in case of shallow indentation (imprint size much smaller than size of autocorrelation function) and in case of deep indentation (several local peaks are covered by the contact area). In the first case, an analytical equation is suggested, which is used to make a multiplicative correction to the mean value of the measured area. The proposed equation is in good agreement with the numerical simulation result. The approach for the second case, which implies the correction for the mean contact depth, was used to correct real experimental value. A qualitative agreement between the corrected and real values of hardness was achieved. It has been shown that the proposed model allows reaching significant improvement of the accuracy of the hardness value measured using instrumented indentation test.

References

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