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Correlation analysis of surface tilt effect on its mechanical properties by nano-indentation

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In this study, finite element analysis and nano-indentation experiments were carried out to investigate the effect of surface tilt on the nanoindentation test results. This paper revealed that standard Oliver-Pharr method underestimated the contact area due to the influence of the tilt condition. Consequently, it is necessary to compensate this difference to ensure that the result is reliable. The finding was verified by the nano-indentation experiments on a sinusoidal surface sample, which is used for the study of correlation between surface topography and its mechanical properties. A corrective action was implemented to compensate the errors by finite element analysis. By eliminating such errors, the study of the relationship between surface topography and mechanical properties was performed and discussed.

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1. Introduction

In the last decade, we witnessed that there was an increasing demand in the capability of accurately measuring surface properties, which include not only geometry parameters, but also surface metallurgy properties such as stiffness, hardness, elastic modulus, adhesion etc.[1-3]. At the same time, more efforts have been made in the area of surface modification to achieve unique surface functions.[4] For example, a variety of unique surface functionalised techniques were applied in industrial applications such as self-cleaning, anti-bacterial, anti-reflection and low friction surfaces.[5-8] It is generally believed that the surface topography affects the surface properties of a bulk material at the macro scale. The impact of a surface geometry on its function is well understood at the macro level. [9, 10] Nevertheless, there are still some ambiguities existed in the relationship between the surface topography and mechanical properties at the micro and nano levels.

Nanoindentation is a common method to obtain the material's mechanical properties at the micro / nanometer level. For example, being capable of measuring the penetration depth in nanometre and the contact force in micro/nano Newton, nanoindentation is an essential technique in the characterisation of the mechanical properties of thin film.[11] However, there are many factors affecting the nano-indentation results.[12] The surface topography of a test sample has a direct impact on the measured nano-hardness values. In addition, very few materials have an atomically flat surface. Generally, the majority of commonly used materials have an unflatten surface, which is formed by undulations and irregularities. Therefore, the surface waviness would impact on the nano-indentation results significantly. In some cases, the sample surface has to be polished prior to nano-indentation experiments. However, for some materials are vulnerable to be damaged by polishing, their mechanical properties would exhibit alteration resulting from work hardening and leading to unreliable measurement results.[13]

Another impact factor is due to sample tilting if assuming the surface is perfectly flat.[14-18] Alignment uncertainties in indentation was investigated by Ellis and Smith through geometry modeling of standard Berkovich, modified Berkovich, and Vickers tips.[14] There was no simulation work done by finite element analysis in their study. In addition, their results were not validated due to the lack of experimental work to investigate alignment uncertainties in indentation. Similar work was carried out by Shi et al., that the effect of tilt on nanoindenation results of fused silica was studied by finite element analysis simulation.[15] A method of correction of error due to sample tilt was developed, while there was no experimental work in the study.

Xu and Li investigated the effect of tilt in nanoindentation test on the fused silica sample using a conical tip. [16] Both finite element analysis simulation and experiments were carried out with tilt at different angles between 1 and 5 degrees. It was found that sample tilt increased the actual contact area, if not considered the effect of tilt, results of hardness and elastic modulus were overestimated. Shahjahan and Hu have recently investigated the angular misalignment of a cylindrical, flat-end tip used in nanoindentation test of low carbon steel AISI 1018.[17] The tilt angle ranged between 0 to 1 degree for indentation testing and finite element analysis simulation. Kashani and Madhavan investigated the effect of surface tilt on nanoindentation by using conical and Berkovich tip in finite element analysis. The results showed the Oliver-Pharr method underestimated the contact area and the mechanical properties were overestimated.[18] The nanoindentation experiment on fused silica by Berkovich tip were performed to compare with simulation results.

The tested and simulated results in above mentioned papers are obtained from assumed smooth and flat surfaces. However, there are some samples with unique surfaces designated to have special topography for specific functions, which are not allowed to be further polished to have a flat/smooth surface. In general, there are always some nano-features existed on prepared samples. Therefore, it is necessary to investigate the tilt effect of these samples on the nano-indentation results and assess the errors associated with the surface topography. Compared to previous studies, the originality and advantage of the current study is that the effect of tilt sample was investigated for the sample with sinusoidal surface, these results can be used for the further research on the correlation between the mechanical properties and surface topography.

In this paper, we present the study on the tilt effect in nanoindentation by applying both finite element analysis simulation and the actual nano-indentation experimentation. The testing system is a home-made tribological probe microscope (TPM), which was designed for localized surface property measurements, including topography, hardness, friction force and elastic modulus.[19-21] In order to study the relationship between the topography and mechanical properties, a sample with standard sinusoidal surface has been selected to be used for nano-indentation by both finite element analysis (FEA) and TPM measurements. Based on the finite element analysis results, the true contact area at each measurement point of the sinusoidal surface was obtained and thus corrections were applied to the raw data obtained by TPM. Finally, the correlation between surface topography and its mechanical properties was carried out after eliminating the potential contact error in the measurements.

2. Methodology

In the process of nano-indentation, the applied force and the depth of penetration are recorded at the same time. The Oliver-Pharr (O-P) method[11] is widely used in the instrumented indentation test to calculate the hardness and elastic modulus. More specifically, the hardness is defined as the ratio between the maximum indented force and the projected contact area.

$$H = \frac{P_{\max}}{A_{cproj}} \tag{1}$$

Where H is hardness, $P_{\max}$ is the maximum indent force and A_{cproj} is the projected contact area. The elastic modulus can also be derived based on the elastic contact theory of Sneddon's analysis[11], which is shown in Eq. (2):

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A_{cproj}}} \tag{2}$$

Where E_r is the reduced elastic modulus, β is a correction factor which is accounted for the tip geometry. S is contact stiffness, which can be derived from the unloading force-depth curve. In the actual experiments, a Berkovich tip was selected, the value of β is 1.072 for Berkovich tip.[22] The deformations of the indenter and the sample are considered in the calculation. Therefore, the reduced modulus can be defined as:

$$\frac{1}{E_r} = \frac{1-v_s^2}{E_s} + \frac{1-v_i^2}{E_i} \tag{3}$$

Where E_S and v_S represent the elastic modulus and Poisson ratio of the testing sample, respectively. E_i and v_i represent the elastic modulus and Poisson rate of the Berkovich tip. The projected area is the major factor in the determination of the mechanical properties by the nanoindentation method. The projected area is calculated based on an analytic function of the contact depth, $A = f(h_C)$. For a Berkovich tip, $A = 24.5h_c^2$. The contact depth is given by the Sneddon equation below[11]:

$$h_c = h_{\max} - \varepsilon\frac{P_{\max}}{S} \tag{4}$$

Where $h_{\max}$ is the depth at the peak load ($P_{\max}$). ε is another correction factor which depends on the tip geometry. The value of ε is set to be 0.75 for Berkovich tip according to O-P method.[11]

3. Nanoindentation on tilt sample

3.1 Finite element simulation

The finite element analysis software ABAQUS 6.14 was used to simulate the tilt situation of a sample under nano-indentation.[23] In this study, a three-dimensional model of a rigid Berkovich tip impressing on a sample with flat surface was developed as shown in Figure 1. The Berkovich tip was defined as an analytical rigid part which is perfectly sharp in a pyramid shape. The tilt angle of the sample with respect to y-axis is between 0 and 2.5 degree as shown in Figure 1(b), the twist angle around y-axis is not considered in this study. The indented sample was modelled as a cube with the dimension of 2×2×2 μm^3. There is no friction considered between the rigid tip and the surface of the sample in this study. To be consistent with the actual experiments, the simulation process was under force control by applying 5mN force on the tip. The material was modelled as an elastic-plastic material with elastic modulus 72 GPa, Poisson ratio 0.17 and yield strength 7.2 GPa.

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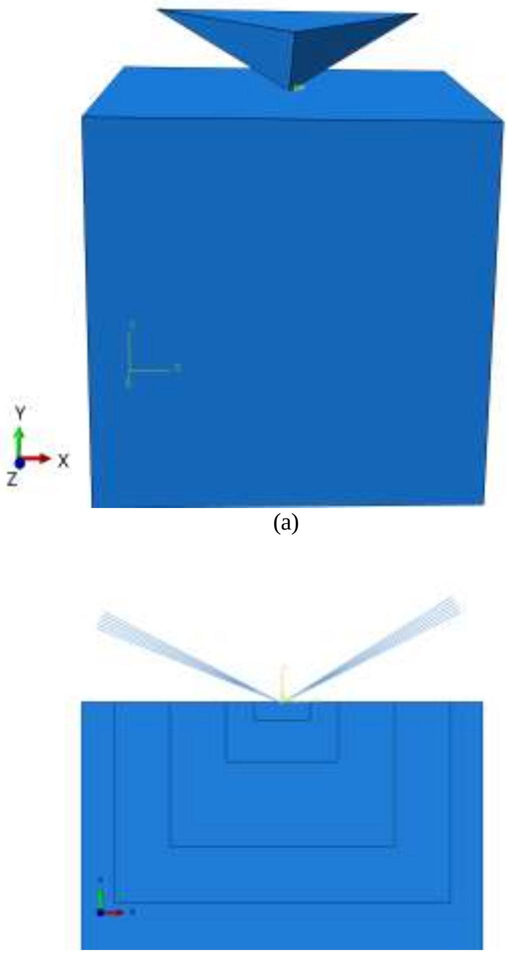


(a)

(b)

Fig. 1 Model of tilt nanoindentation on a flat sample in Abaqus: (a) 3D view; (b) Cross section view

The relationship between the applied force and penetration depth is demonstrated by force-depth (P-h) curve, which can be obtained from the reaction force of the tip and the vertical displacement of the contact point of the sample. The reaction force and displacement of sample can be obtained by Abaqus software when simulation completed.

In order to have a high efficiency calculation, a progressive coarse mesh was set in the area far from the contact zone. To obtain an accurate result, finer mesh was employed in the contact region in Figure 2. For the sensitivity analysis of this finite element model, the primary factors of this model are the selection of mesh element and the element size. The simulation sample was modeled as C3D8, C3D8R and C3D8I elements.[23] The proper element size was determined by convergence study of the contact area obtained by finite element analysis.

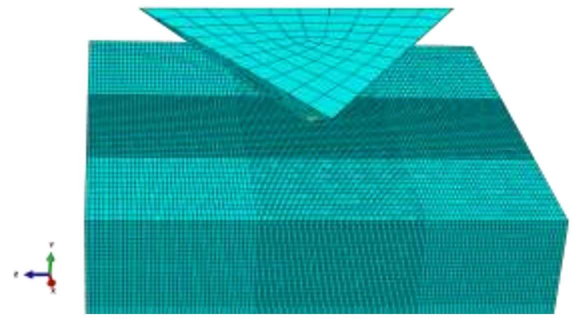


Fig. 2 Mesh of the nanoindentation sample

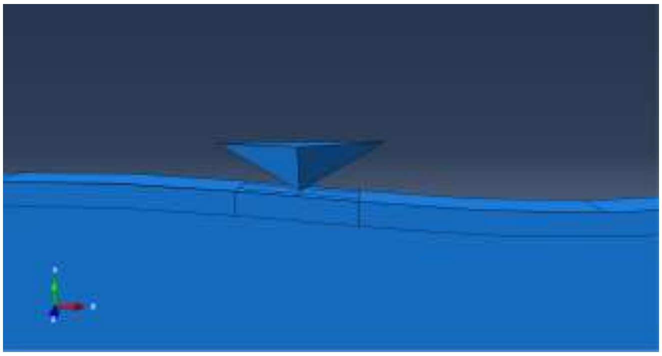
3.2 Nano-indentation experiments on a sample with flat surface

The actual experiment was performed on a flat sample, which is a round disk in 20 mm diameter with 2 mm thickness. The material of this sample is fused silica, which is commonly used as a calibration material for nanoindentation test. There is no further treatment of the sample before the experiment. The roughness of this sample was measured by using a white light interferometry. The average roughness of the sample is 0.015 μm. The facility used for nano-indentation test is a unique homemade equipment, Tribological Probe Microscopy, which has successfully been used to measure the mechanical properties of a variety of materials in previous studies.[19-21] This testing system has been calibrated according to ISO 14577.[24] An exclusive sample holder that can tilt the sample horizontally to maximum 2.5 degrees was made for this test. All the experiments were performed under force control with a diamond Berkovich tip to indent the sample. The testing facility was calibrated prior to the tilt indentation. With a careful operation, five indents were conducted at each tilt angle of 0, 0.5, 1, 1.5, 2 and 2.5 degrees under a constant peak force of 5 mN.

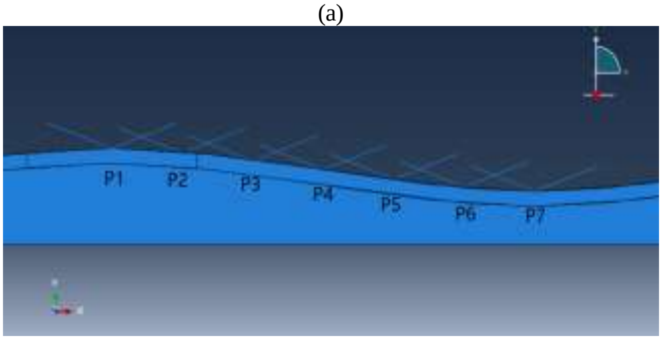
4. Nano-indentation on the sample with sinusoidal surface

4.1 Finite element simulation

A 3D finite element model of nano-indentation on a sinusoidal surface sample was established by using software ABAQUS 6.14.[23] The indentation tip was modelled as a sharp Berkovich tip. The surface of the tip was set as an analytical rigid part. The material of the sample is set as elastic-plastic model with elastic modulus 72 GPa, Poisson ratio 0.17 and yield strength 7.2 GPa. The local variation of the sinusoidal surface is used to investigate the tilt effect, thus the nano-indentation was performed at different positions (from P1 to P7) on this sample, as shown in Figure 3.



(a)



(b)

Fig. 3 FEA simulation of nanoindentation on a sinusoidal surface sample: (a) 3D view; (b) Cross-section view

Therefore, there is no tilt angle at positions P1 and P7 as shown in Figure 3 (b) due to they are either the top or the bottom of the sinusoidal curve, while the tilt effect at position P4 is maximum. In order to simplify the situation, the friction between the tip and the surface is not considered. The indentation was performed under a peak force of 100 mN on each position. The P-h curve was directly obtained from the applied force and the output of the deformation depth.

4.2 Experiment on standard sample with sinusoidal surface

The sample used in this experiment has a standard sinusoidal surface, which is made from pure electroformed nickel with a hard top film of nickel-boron. This sample was measured under a white light interferometry to obtain the surface topography. The experiment was performed under the same settings as in section 3.2, but without the tilt holder. The multi-function measurements include topography, hardness and elastic modulus were carried out by TPM with an initial scanning force of 1 mN and a peak force of 100 mN over an area of 100×100 μm^2. Due to the unique function of TPM, it can measure both the topography and mechanical properties at the same time, this allows us to investigate the correlation between the mechanical properties and topography of the sinusoidal sample.

5. Results and discussion

Figure 4 shows the P-h curves obtained from FEA of the Berkovich tip indentations with a tilt angle between 0 and 2.5 degrees on a flat sample. It is clearly shown that under the same peak force of 5 mN, the penetrated depth decreases with the increasing of the tilt angle. In other words, under the same applied force, no tilt indentation produces deeper penetration depth than any tilt indentation.

The corresponding experimental results under the tilt indentation are shown in Figure 5 and it can be seen that the trend is very similar compared to the FEA simulations in Figure 4. The P-h curves obtained from actual experiments show that the penetration depth is decreasing with the increasing of the tilt angle under the same peak force of 5mN.

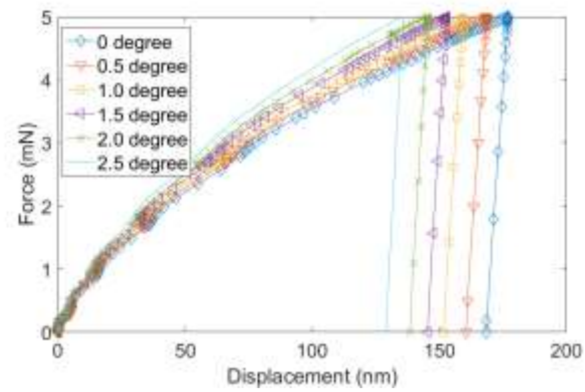


Fig. 4 FEA simulation result of P-h curves under different tilt degree

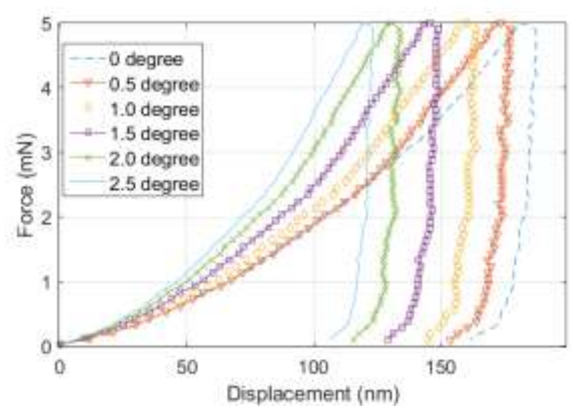


Fig. 5 P-h curves of nano-indentation experiments

The contact area of each indentation can be derived by using the O-P method which is based on the loading-unloading curves.[11] Then the mechanical properties of the test sample, such as hardness and elastic modulus can be calculated according to Eq. (1) to Eq. (4). In addition, the contact area was computed from the finite element simulation outputs under each tilt angle. The projected contact area can now be calculated by using the Eq. (5), which is projected in the plane perpendicular to the vertical axis.

$$A_{cproj} = A_c^{FEA} \cdot \sin\theta \quad (5)$$

Where A_c^{FEA} is the result of contact area in FEA modelling, θ =65.30 for a Berkovich indenter. A_{cproj} is the projected contact area. The results of projected contact area under tilt condition can be obtained by the O-P method and FEA simulation outputs. The contact area computed by FEA decreases as the function of tilt angle from 0 to 2.5. This is because a constant indentation peak force of 5mN was applied for each tilt angle. According to Figure 4, the maximum penetration depth decreases as the function of tilt angle, more tilt less penetration depth under the same indentation force.

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For the sensitivity analysis of finite element model, the elements used in the simulation were fully integrated with eight-node hexahedral element (C3D8), reduced eight-node hexahedral element (C3D8I), incompatible eight-node hexahedral element (C3D8I). Fully integrated eight-node hexahedral element was used in many other studies, [16,18] however the limitation of fully integrated element is shear locking when subject to bending. Element C3D8R and C3D8L are able to improve the behavior when subject to bending. The results of model meshed by C3D8 is very close to model with C3D8I (less than 0.3% in contact area deviation). The results by C3D8R exhibited 4.0% higher value in contact area. Studied was also performed to analyse the sensitivity of model to element mesh size. A convergency study was undertaken until obtained FEA output parameters are stable, e.g. deviation of contact area is less 1%. The threshold of mesh size adjacent to the contact area is 10nm.

The comparison of the relationship between the contact area and tilt angle is shown in Figure 6, which is calculated based on the curves in Figure 4. Although the trends of two curves are similar, the contact area decreased as a function of increased tilt angle from 0 to 2.5 degrees. It was clearly that O-P method underestimated the contact area compared to the FEA simulation results. For instance, under 2.5 degrees tilt condition, there is only 2% deduction based on FEA output while O-P method presents 10% reduction.

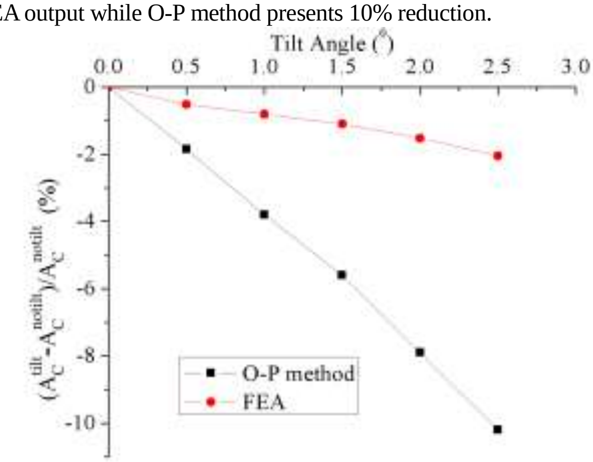

Fig. 6 The variation of the contact area due to tilt by both O-P method and FEA simulation

Due to the difference in the projected contact area, the hardness (H) and elastic modulus (E) are much varied against the tilt angles. Figure 7 shows that the standard O-P method gives underestimated contact areas which result in overestimating the hardness (H) and elastic modulus (E) values. Based on the projected contact area calculated from FEA outputs, H and E remain less dependent on the tilt angle. The difference between these two approaches goes up as the tilt angle increases. In the case of maximum tilt of 2.5 degrees, the standard O-P method overestimated the hardness and elastic modulus for 11% and 4%, respectively.

Similar results were also found in nano-indentation experiments. Based on the standard O-P method, the measured results show a clear overestimation of hardness values by 10% and elastic modulus by 5%. By applying the corresponding correction factors, which are derived from the FEA method as discussed above, the values were down to 2% for hardness and 1% for elastic modulus. Therefore, it is demonstrated that by applying the FEA correction for the contact area obtained from the measurements, the errors due to tilt indentation can be minimised.

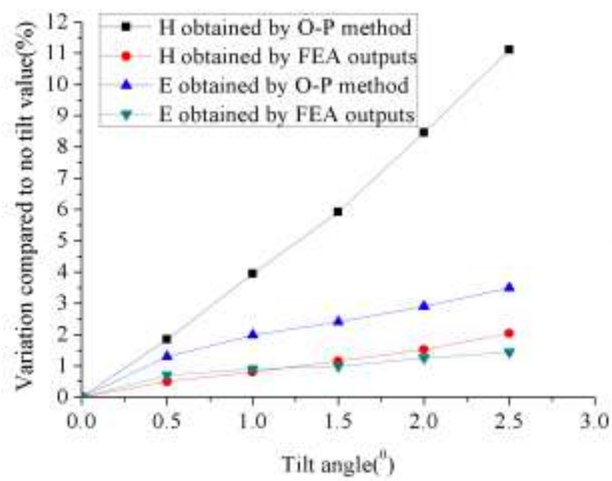

Fig. 7 Comparison of E and H values from FEA and O-P method at different tilt angles

For the indentation on a sample with sinusoidal surface, which naturally exhibits undulation even if under perfectly perpendicular alignment, the results show a similar tendency compared to the tilt indentation on flat sample. The FEA simulation of nanoindentation on a sinusoidal surface was carried out. The simulation outputs of P-h curves are shown in Figure 8. Under the same applied force, the penetration depth varied at different positions of the sample. These variations in P-h curves result in the variation in nanoindentation results. Figure 9 shows the calculated E and H values based on the standard O-P method and FEA simulation outputs. At the maximum slope of position P4, the hardness and Young's modulus values are the largest compared to values of other positions. By applying the FEA correction, the variations in H and E reduce 13% and 9%, respectively. It is shown that the O-P method overestimates the mechanical properties, especially more at the positions from P2 to P6. Such potential errors from tilt indentation could be minimised by the FEA corrections, if the tilt angle is known.

Consequently, it is necessary to modify the results measured by TPM on the nickel specimen with a sinusoidal surface. The sinusoidal surface parameters of the nickel sample are listed in Table 1 from the surface measurements carried out by WYKO-NT2000 and TPM. In addition, the mapping images of topography, hardness and elastic modulus of the sinusoidal sample are shown in Figure 10.

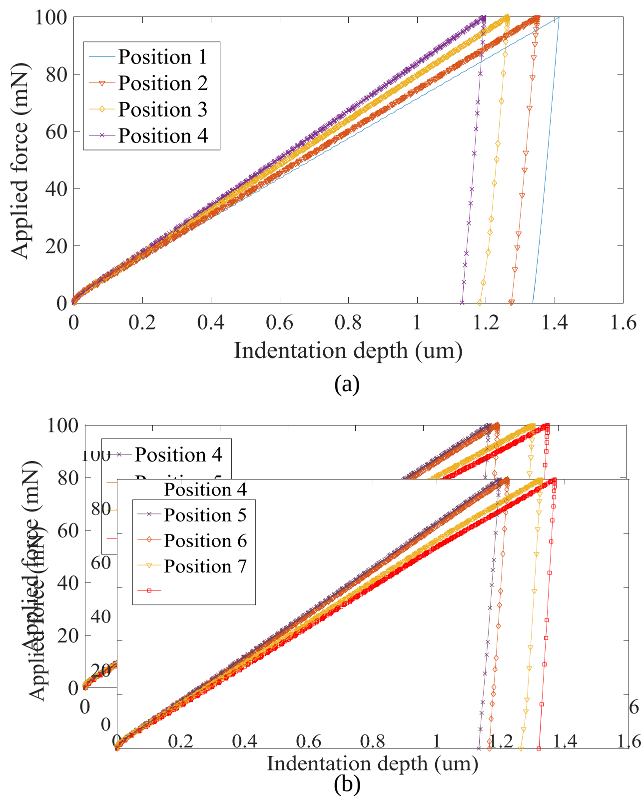

Fig. 8 P-h curves obtained at different position of the sinusoidal surface from FEA as illustrated in Fig. 3: (a) Positions 1 to 4; (b) Positions 4 to 7

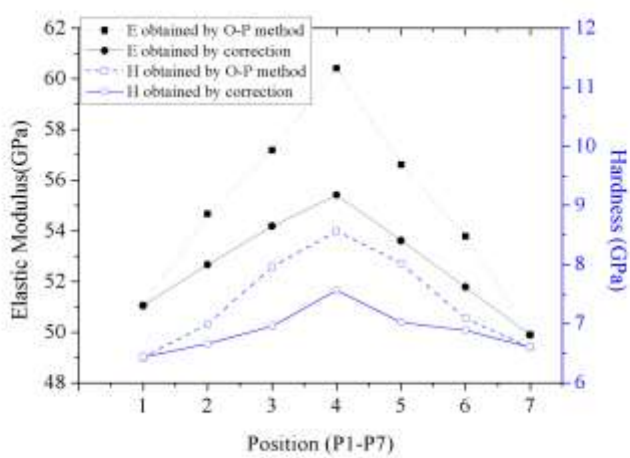

Fig. 9 Comparison of E and H obtained by the O-P method and FEA correction for indentation at different positions of a sinusoidal surface

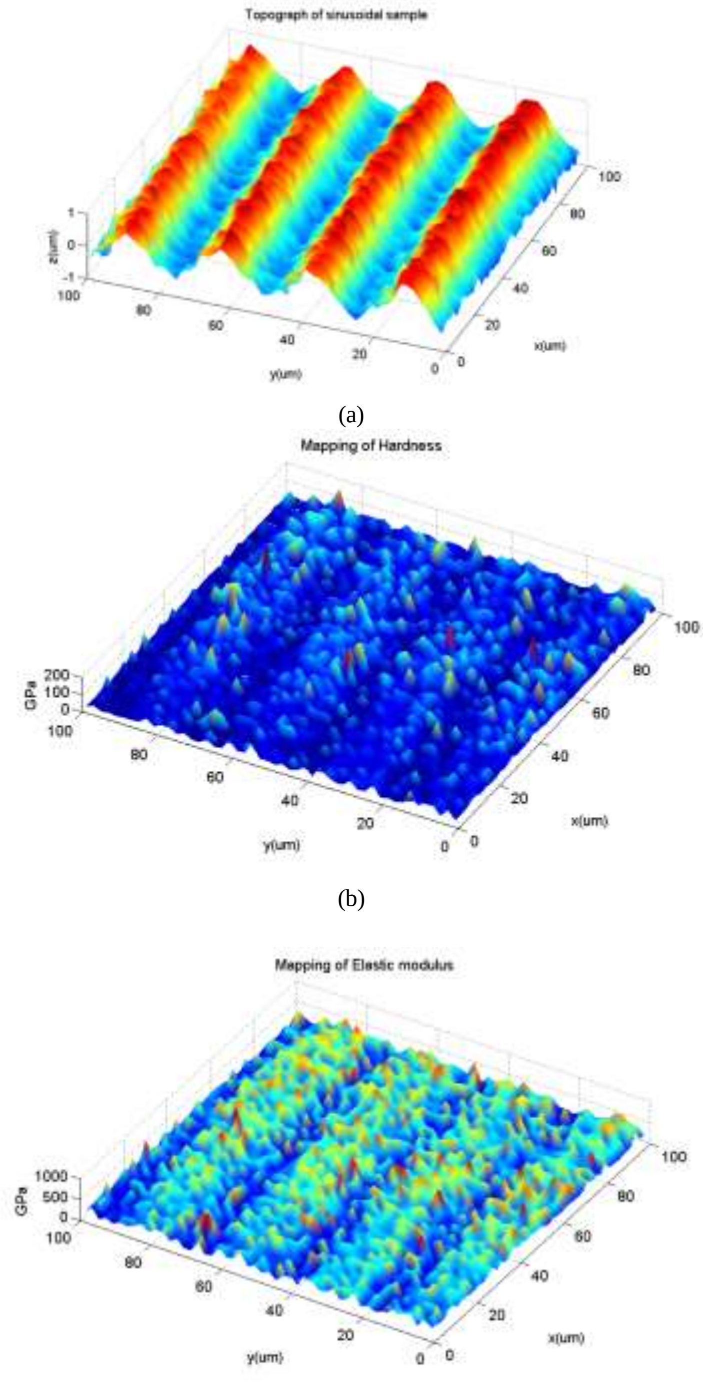

Fig. 10 Mapping results of mechanical properties by TPM: (a) topography; (b) hardness; (c) elastic modulus

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Table 1 Surface parameters of sinusoidal sample measured by WYKO –NT2000 and TPM

	WYKO NT2000	TPM
R_a (μm)	0.351	0.325
R_q (μm)	0.388	0.370
R_t (μm)	1.50	1.20
S_m (μm)	25.0	25.1
E (GPa)		225.3
H (GPa)		27.3

Three images obtained by TPM in one set-up and originally the efforts have been made to see how surface topography affects the mechanical properties. Obviously, the local slope of the sinusoidal surface acts like tilt effect on nanoindentation results. As the measurements were made point-by-point at the same time, the images thus obtained can be used to investigate the correlation between the surface topography and mechanical properties.[25] According to the FEA simulation results of sinusoidal surface, the original results of mechanical properties are overestimated in some regions of this periodical surface. Consequently, the correction of original results needed to remove the potential errors resulting from the measurements of undulate surface. Figure 11 shows the averaged surface profile, average hardness profile and the corrected hardness profile over an area of 100×100 μm^2. It is interesting to find that there is a clear difference between both sides of sinusoidal surface in hardness, which results from the rotationally asymmetric of Berkovich tip that leading to the different in contact areas on both sides.

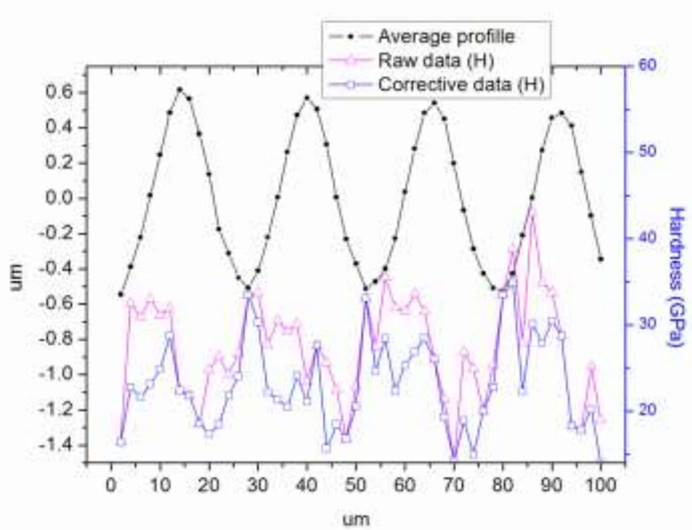

Fig. 11 Averaged topography, hardness before and after correction for the sinusoidal sample

It is demonstrated that the effect of topography on its mechanical properties is less than expected. The cross correlation between topography and mechanical properties were calculated. On the basis of the raw data, the correlation value between topography and hardness is -0.38, while after correction, this result was down to -0.25. The negative result of correlation factor was also found on other types of material according to previous work.[25] The original averaged hardness is 27.3 GPa with a standard deviation of 6.3 GPa for the sample. After correction, the mean value was down to 23.1 GPa and the standard deviation decreased to 4.1 GPa. Similar results were found in the elastic modulus. These findings indicate that although there is a negative relationship between the surface topography and hardness, the level of their relations was not as high as expected before. The corrected results reveal that the waviness surface still has an impact on its mechanical properties; the error resulting from the tilt indentation can be reduced by means of FEA corrections.

6. Conclusions

This study has explored the impact of surface tilt on nano-indentation results. FEA simulation and nano-indentation experiments have been conducted to demonstrate that the standard O-P method underestimated the contact area which leads to an overestimation of the elastic modulus and hardness of test samples. The result shows that the errors caused by 2.5 degrees tilt are 4% in elastic modulus and 11% in hardness. Similar observation has been found in the study of a standard sample with sinusoidal surface. Corrections to the preliminary results based on FEA simulation were carried out to compensate the errors due to tilt impact on the nanoindentation results. By doing this, the correlation factor between the sinusoidal surface and its hardness was altered from -0.38 to -0.25. Moreover, the average and standard deviation of hardness also decreased from 27.3±6.3 GPa to 23.1±4.1 GPa. This conclusion reveals that the degree of surface waviness impact on its mechanical properties is not as great as expected before.

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