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BLUNTNESS MEASUREMENT OF A BERKOVICH INDENTER

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Indenter blunting is inevitable in nanoindentations and results in unexpected contact properties. Both relaxation of the indentation size effects and deepening of the substrate effects under a blunted indenter cause a change of the bathtub-shaped hardness with indentation depth in a soft film on hard substrate. Thus an identification of three-dimensional morphology of an indenter apex is necessary for precise measurements of hardness in thin films. We observed an actual Berkovich indenter using an atomic force microscope (AFM). Through a quantitative analysis on the AFM image, data pairs of contact area versus contact depth were obtained; curvature radius of the apex was estimated by searching a sphere well-fitted to the indenter apex morphology. The estimated curvature radius and blunted height were 1043.9±50.9 nm and 44.4 nm, respectively. By comparing with the result from the modified Kick's law, both blunted heights were comparable each other within a 7 nm difference. This confirms validity of the direct observation method with the AFM.

Keywords: Nanoindentation; Atomic force microscopy (AFM); Berkovich indenter; Apex bluntness.

1. Introduction

A nanoindentation test provides real-time, load-indentation depth data while the indentation is in progress.[1,2] The nanoindentation hardness is defined by L_{max}/A_C. Here, A_C is the contact area under the peak load, L_{max}. Oliver and Pharr[2] proposed a derivation method of the contact depth, h_C from the nanoindentation unloading curve and this contact depth, h_C can be converted to the contact area, A_C based on the indenter geometry; a Berkovich indenter has a 65.3° nominal angle between faces and principal axis and it holds an indenter geometry function, $A_C = 24.56\ h_C^2$.

However, an actual Berkovich indenter has a degree of bluntness. A sharp indenter forms stress singularity and it accelerates local wear of the indenter apex. According to a previous research[3], the blunted indenter reduces the hardness variation at shallow indentation regime. The relaxation of the indentation size effects modeled by the strain gradient plasticity[4] was explained by a sparse distribution of the geometrically necessary dislocations[3]. In addition, a severe increment of hardness is reported at deep indentations comparable to the film thickness and this is ascribed to expanded deformations of the hard substrate beneath the blunted indenter[5]. Thus many researchers measured the indenter morphologies using an atomic force microscope[6], confocal microscope[7] and optical interferometer[8]. However, these experiments are related to macroindenters and in-depth observations on the apex bluntness are still insufficient. Recently, Park et al.[5] proposed a prediction method of the indenter bluntness by fitting the indentation loading curve with the modified Kick's law.

Thus we observed and analyzed three-dimensional apex morphology of a Berkovich indenter with an atomic force microscope. Three-dimensional information of the apex geometry was recorded in an ASCII data file and analyzed through a series of image analyses; curvature radius and blunted height of the indenter were estimated and compared with the results from Park et al.'s bluntness prediction.[5]

2. Experimental Procedures

Three-dimensional observation of a Berkovich indenter was done with an atomic force microscope (XE-100 model, Park Systems Corp., Suwon, Korea). A silicon nitride cantilever was used as a contact mode scanning with a probing load of 100 nN. The AFM image was calibrated with in-plane and out-of-plane standard blocks. The in-plane standard block (TGX01 model, MikroMasch, Tallinn, Estonia) has a chess pattern with a periodic wavelength of 3000±8 nm. The out-of-plane standard block (Step Height CRM 207-04-004, KRISS, Daejeon, Korea) has a step height of 541±5 nm.

The Berkovich indenter (Micro Star Technologies, TX, USA) with a long-term usage was cleaned with a cotton swab wetted with ethyl-alcohol and prepared for the AFM observations. For the indenter apex, scanning area was estimated at 5×5 μm^2 and scanning speed was controlled at less than 1.0 μm/s. In addition, the nanoindentation tests were carried out on a polished fused silica with a Nanoindenter XP (MTS Corp., TN, USA). The peak load and loading rate were 200 mN and 0.05 /s, respectively.

3. Results and Discussion

3.1. *Direct observation of the indenter apex*

AFM images were calibrated using two standard blocks; calibration ratios of patterned area to scanned one were estimated at 1.07 and 1.19 for the in-plane and out-of-plane blocks, respectively. Raw ASCII file converted from the AFM image was calibrated by multiplying the calibration ratios. Apex morphology of the indenter is shown in Fig. 1.

Bluntness was clearly observed due to the accumulative wear during repetitive nanoindentations. The blunted regime changes gradually into the pyramidal regime with an increase of the indentation depth. When an indenter penetrates into specimen without surface irregularities such as pile-up and sink-in, the indentation depth, h becomes the contact depth, h_C and corresponding contact area, A_C can be measured by superposing an in-plane cutting plane on the three-dimensional morphology of the observed apex. A one-to-one relationship is constructed from the plot of A_C versus h_C shown in Fig. 2. If the empirical relationship at shallow indentations in Fig. 2 is fitted to the geometry of a sphere, the curvature radius, R of the indenter apex can be estimated as shown in Fig. 3. If the blunted indenter is treated as a sphero-conical shape in the inset of Fig 3, the blunted height, h_b can be estimated from R and the indenter semi-apex angle, θ. R and h_b were 1043.9±50.9 nm and 44.4 nm, respectively, when the measured value of θ was 73.4° for the indenter.

3.2. *Bluntness prediction from the nanoindentation loading curve*

Due to the bluntness, the nanoindentation curve shifts leftwards and its amount is the blunted height. Thus the Kick's law[9] is modified into $L=K(h+h_b)^2$, where K is the fitting constant. If square roots are taken for both sides of the modified Kick's law, L can be expressed by a linear proportional relationship with h; the proportional slope is $\sqrt{K}$ and the intercept along the Y-axis is $h_b\sqrt{K}$.[5] Linear proportional graph is plotted in Fig. 4; whole plots show straight lines but initial contact regimes are deviated from the straight lines due to the effects of the blunted apexes. From the proportional slope and the Y-axis intercept, the blunted height was estimated at 37.4±1.8 nm for the indenter. A comparison of blunted heights estimated by both direct observation and prediction method[5] supports validities of both approaches. As discussed in the previous study[5], the measured bluntness can be used for the correction of the nanoindentation curves of ultrathin films. It means that an estimation of the indenter bluntness must be carried out before nanoindentation tests on ultrathin films as one of calibration procedures.

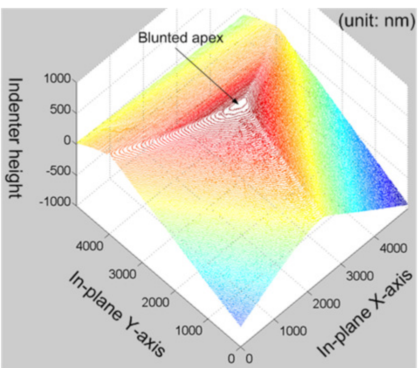

Fig. 1. Three-dimensional morphology of the Berkovich indenter.

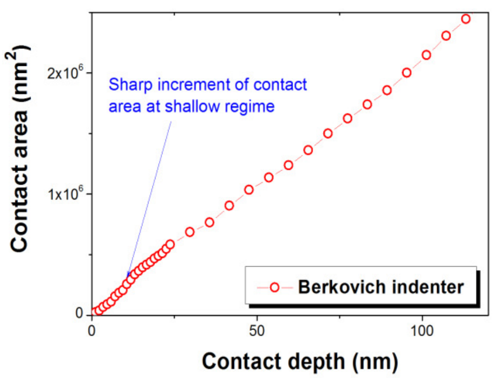

Fig. 2. Variation of the contact area with an increase of the contact depth.

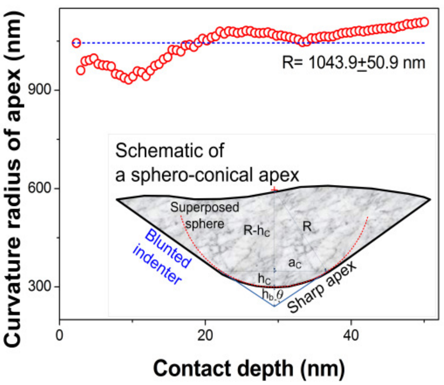

Fig. 3. Curvature radius of the Berkovich apex and schematic diagram of the indenter.

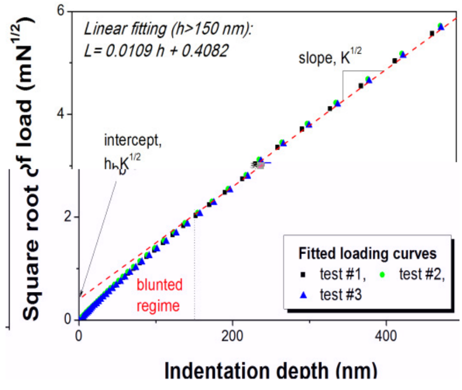

Fig. 4. Linear line fitting for the nanoindentation loading curves obtained from fused silica.

4. Conclusions

We observed a Berkovich indenter with an atomic force microscope in order to estimate its bluntness. Firstly dimensions of the AFM image were calibrated with two patterned, standard blocks. Then precise image analyses were carried out for the three-dimensional apex; data pairs between the contact area versus the contact depth were obtained and curvature radius and blunted height of the apex were determined by fitting the blunted Berkovich morphology into a sphero-conical geometry. The blunted height was 44.4 nm comparable with the bluntness value, 37.4±1.8 nm predicted from the nanoindentation loading curve of fused silica.

Acknowledgments

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