Influence of surface-roughness on indentation size effect

Ju-Young Kim [a], Seung-Kyun Kang [a], Jung-Jun Lee [a], Jae-il Jang [b,*], Yun-Hee Lee [c], Dongil Kwon [a]

[a] *Department of Materials Science and Engineering, Seoul National University, Seoul 151-744, Republic of Korea*
[b] *Division of Materials Science and Engineering, Hanyang University, Seoul 133-791, Republic of Korea*
[c] *Division of Metrology for Quality Life, Korea Research Institute of Standards and Science, Daejeon 305-340, Republic of Korea*

Abstract

During nanoindentation of a material with a naturally rough-surface, a flattening of the rough-surface is additionally accomplished compared to nanoindentation on a flat surface. By separating analytically the work expended to flatten the rough-surface and to deform the flattened surface, we develop here a new rough-surface indentation size effect (ISE) model. This new model is applied to nanoindentation results for three Ni samples of different surface-roughness and the applicability of the model is discussed in terms of a critical contact depth for the surface-roughness effect on ISE.
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1. Introduction

Over the past decades, advances in nanoindentation techniques along with the development of commercial equipment have made it possible to explore the mechanical properties and behavior of very small volumes of material, as reviewed by several researchers [1–7]. From extensive research through nanoindentation experiments, it is now generally accepted that the indentation hardness measured even with a geometrically self-similar pyramidal indenter (e.g., the commonly used Berkovich indenter) increases with decreasing indentation depth or force, which is the so-called indentation size effect (ISE) [8–31].

Based on Ashby's suggestion that geometrically necessary dislocations (GNDs) would increase the strength in bending or flat-punch indentation [32], many early works on the ISE [8–11] proposed a possible relationship between the GNDs and the ISE. In 1998, the most popular mechanism-based model of the ISE phenomena was established by Nix and Gao [12], who considered the density of GNDs

generated by a geometrically self-similar sharp indenter together with a Taylor's dislocation model [33]. In the Nix–Gao model, the relation between the indentation hardness (H) and the indentation depth (h) can be simply described as:

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}}, \tag{1}$$

where h^* is a characteristic length depending on both the indented material and the indenter angle and H_0 is the macroscopic indentation hardness (when h is much greater than h^*). Since the linear relation between $(H)^2$ and $(1/h)$ in Eq. (1) successfully predicted the experimental indentation hardness data, the Nix–Gao model has been applied extensively (sometimes with minor revisions) and Swadener et al. extended it to a spherical indenter by assuming a parabolic geometry of the indenter [19].

However, it has been found from further research that at very shallow indentation depth (typically <100 nm), nanoindentation hardness data can deviate significantly from the predictions of the Nix–Gao model. It was suggested that this deviation at small indentation depths might be due to the inherent response of materials during nanoindentation

(Peierls stress, storage volume for GNDs and so on [17,23,28]) as well as several extrinsic factors such as blunt tip on a sharp indenter, surface-roughness, oxide layer, chemical contamination and work-hardened layers [16,22,26]. Among the extrinsic factors, some degree of surface-roughness is almost unavoidable in nanoindentation experiments [34] and thus has been of interest. Bobji and Biswas [35] demonstrated via computational simulations that surface-roughness has a significant effect on hardness. Gerberich et al. [36] divided the work done by an applied indentation force into surface work and volume work and included the surface-roughness effect in the surface work. Most recently, Zhang et al. [24] modified Eq. (1) of the Nix–Gao model and clearly demonstrated the effect of surface-roughness on the ISE by assuming flattening of the indented rough-surface by fully plastic deformations:

$$H = H_0 \sqrt{1 + \frac{h^*}{h} + \frac{2e_\mathrm{c} + gf_\mathrm{s}}{h}}, \qquad (2)$$

where e_c is the dissipation energy per contact area due to plastic deformation, g is a geometric constant and f_s is the thermodynamic surface stress. However, from a practical viewpoint, some difficulties can arise in applying this bearing ratio model because e_c and f_s are hard to measure experimentally. It is thus still desirable to derive a more easily applicable relation between surface-roughness and ISE.

With this in mind, here we propose a new rough-surface ISE model. During nanoindentation, it is plausible that the material surface in contact with the indenter, regardless of its original roughness, becomes topographically smooth. Thus, material deformation by nanoindentation is accomplished by the combination of two simpler procedures: flattening of the indented rough-surface and deformation by nanoindentation of the flattened surface. The dissipated work terms for each step were derived analytically and their ratios are presented with the contact depth and ISE characteristic values. Based on the separation of the dissipated work terms, a new rough-surface ISE model is developed and its validity is experimentally examined. Our ultimate goal is to characterize the ISE by interpreting the nanoindentation hardness at shallow depths excluding the surface-roughness effect, which may be a principal extrinsic ISE factor.

2. Experiments

The surfaces of three 99.99% pure Ni samples were carefully polished with 0.05, 1 and 5 μm alumina powder intentionally to control the average surface-roughness R_a. The values of R_a were measured using an XE-100 (PSIA Inc., Suwon, Korea) atomic force microscope (AFM). The scan area was 3×3 μm close to the residual indentation impression area. Nanoindentation experiments were conducted using a Triboindenter (Hysitron Inc., Minneapolis, MN) with a three-sided pyramidal Berkovich

diamond indenter. The maximum indentation force P_max was 5 mN and the loading and unloading rate dP/dt was 300 μN/s. The change in hardness with indentation depth was measured by partial unloading at six different indentation depths. Directly after the indentation experiments, the geometrical profiles of the residual indentation impressions were measured using the Triboindenter's AFM function, from which the final pile-up height h_pile around the impression was determined. Since the measured h_pile is valid only for the final unloading, the values of h_pile at each partial unloading were estimated by assuming that the ratio of h_pile to the maximum indentation depth, h_max, is approximately constant and independent of indentation depth [13].

3. Results and discussion

3.1. Measurement of surface-roughness and hardness

Fig. 1 shows the typical surface morphology and the average surface-roughness, R_a, with standard deviation measured by AFM. The parallel scratches on the surface were caused by mechanical polishing: the Ni sample polished with coarser alumina powder shows the greater roughness (e.g., $R_\mathrm{a} = 8.65 \pm 0.73$ nm and 3.22 ± 0.33 nm for 5 μm and 1 μm powder, respectively). The surfaces polished with 0.05 μm alumina powder are so close to flat ($R_\mathrm{a} = 0.44 \pm 0.07$ nm) that they can be assumed to be flat surfaces. Note that R_a is not equivalent to the mean value of the maximum height difference between the top of peak and the bottom of valley in the surface (R_max designated in ISO 4287 [37]); this maximum height difference measured experimentally in the present work was several times R_a. The detailed procedure for determining R_a is described in ISO 4287 [37] (see also the authors' previous study [34]). Fig. 2 shows the statistical distributions of surface heights, which exhibit a normal distribution regardless of average surface-roughness.

Fig. 3 shows the change in hardness H ($=P_\mathrm{max}/A_\mathrm{c}$, where A_c is contact area) as the contact depth h_c increases. This contact depth h_c was derived by adding h_pile (measured by AFM) to the conventional contact depth in the Oliver–Pharr method [38], i.e., $h_\mathrm{c} = h_\mathrm{max} - h_\mathrm{d} + h_\mathrm{pile}$, where h_d is the elastic deflection depth. The contact area A_c was then determined by inputting this h_c into the area function obtained from preliminary nanoindentation experiments on a fused quartz standard specimen [38]. In Fig. 3, the hardness values are clearly dependent on surface-roughness at shallow contact depths (less than about 100 nm), while they are similar at larger contact depths (greater than about 100 nm). Considering the pile-up height, the indentation depth h in Eq. (1) can be replaced by the contact depth h_c defined above, i.e.,

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h_\mathrm{c}}}. \qquad (3)$$