Surface roughness effect in instrumented indentation: A simple contact depth model and its verification

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Since in instrumented indentation the contact area is indirectly measured from the contact depth, the natural and unavoidable roughness of real surfaces can induce some errors in determining the contact area and thus in calculating hardness and Young's modulus. To alleviate these possible errors and evaluate mechanical properties more precisely, here a simple contact model that takes into account the surface roughness is proposed. A series of instrumented indentations were made on W and Ni samples whose surface roughness is intentionally controlled, and the results are discussed in terms of the proposed model.

Over the past two decades, the instrumented indentation technique (especially nanoindentation) has advanced rapidly, and it is now one of the most powerful tools for evaluating small-scale mechanical properties and deformation behaviors.[1,2] In performing instrumented indentation tests to estimate hardness and Young's modulus of small volumes (typically according to the Oliver-Pharr method),[3] a fundamental advantage is that measuring the residual impression area by an imaging tool is unnecessary; rather, the contact depth, h_c, which is a parameter quantifying the contact morphology in the loaded state, is determined by analysis of the indentation load (P)-depth (h) curve and is used to indirectly measure the contact area, A_c.

Since the contact area is determined from the contact depth, the natural and unavoidable roughness of real surfaces can induce some significant errors in assessing hardness and Young's modulus by nanoindentation. The influence of surface roughness can be critical when it cannot be controlled intentionally [e.g., in very thin films, microelectromechanical systems (MEMS), and bio-tissues][4–7] or when the roughness after surface treatment is still nonnegligible compared with the maximum indentation depth, h_{max}. Although a lot of studies have reported the existence of surface roughness effect, limited efforts have been made to quantify the effect on contact depth determination. The purpose of this article is to propose a simple contact depth model that takes into consideration the surface roughness effect and thus makes possible more precise determination of hardness and Young's modulus through instrumented indentation.

To avoid difficulties in intentionally controlling the surface of nanoindentation samples[6] and to observe the roughness effects more clearly on a larger scale, in the present study, instrumented microindentation experiments instead of nanoindentations were performed by instrumented indentation equipment AIS 3000 (Frontics Inc., Seoul, Korea) with a four-sided pyramidal Vickers diamond indenter. Depth-controlled indentations were made on both 99.99% Ni and 99.9% W samples whose surfaces were mechanically polished with diamond paste of 9, 6, 3, 1, and 0.25 μm, respectively, and thus had different roughness. The maximum indentation depth (h_{max}) was set as 50 μm, which was recognized by the instrumented indentation equipment. Before and after indentation, the average surface roughness and the height of pile-up/sink-in were measured by optical profiler with vertical resolution 0.1 nm.

The present work began by taking the possible contact morphology into consideration. During indentation, it is plausible that the material surface in contact with the

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indenter becomes topographically smooth, regardless of its original roughness.[4] In this case, one can consider the indentation process as consisting of two steps: in the first, the rough surface inside the projected contact area (A_c) is flattened, and in the second, the ideally flattened surface is deformed by indentation. In the first step, the asperities inside A_c are deformed perfectly plastically so that the peaks of the asperities flow down to fill the valleys.[8] Zhao et al.[9] showed that this fully plastic deformation of the asperities occurs at the very early stage of contact. Since the radius of indenter tip is usually much greater than those of the asperities, initial contact is likely to occur around the peak of an asperity.[10,11] The height of the material surface, which is the starting point of penetration depth by indentation, is defined in this study as "reference height." If the rough surface inside A_c becomes smooth during the loading sequence, the mean height, δ_m, of the original asperities, rather than their representative peak height, δ_o, can be taken as the reference height (see a schematic illustration in Fig. 1). With this in mind, we tried to define the difference between δ_o and δ_m to determine the contact depth precisely.

The realistic description of a rough surface in mathematical form is based on the assumption that surface heights follow a normal distribution.[12] One can determine bounds of the distribution that, in some probabilistic sense, cover the individual height values, of which the upper bound can be considered as δ_o. Clearly, a bound that covers the middle 95% of the height distributions is given by $\mu \pm 1.96 \times \sigma$ where μ is the mean value and σ is standard deviation (sometimes called tolerance interval).[13] Thus, the difference between δ_o and δ_m is assumed to be $1.96 \times \sigma$. For a normal height distribution, the relation between the standard deviation and the average surface roughness, R_a, can be expressed as $\sigma = \sqrt{\pi/2} \times R_a$,[12] and accordingly the difference between the δ_o and δ_m is $1.96 \times (\pi/2)^{0.5}$ times R_a (i.e., $2.46 \times R_a$). Therefore, if the flattening behavior of a rough surface is considered in arriving at roughness-independent mechanical properties, the depth $2.46 \times R_a$ should be subtracted from the indentation depth recorded by the instrumented indentation equipment.

In instrumented indentation tests, hardness is defined as $H = P_{max}/A_c$ and the reduced elastic modulus is estimated by $E_r = \sqrt{\pi}S/2\beta\sqrt{A_c}$, where P_{max} is the maximum load, A_c is contact area, S is stiffness (determined by the initial slope of the unloading curve), and β is a correlation factor (1.012 for Vickers indentation).[3,14] Since A_c is about 24.5 times square of contact depth (h_c^2), precisely determining the contact depth is very important in arriving at accurate H and E. By modifying the height for pile-up/sink-in, which corresponds to the plastic deformation in the loaded state,[15] based on the Oliver-Pharr method, the contact depth is written as

$$h_c = h_{max} - h_d + h_{pile} \quad , \tag{1}$$

where h_{max} is the maximum indentation depth, h_d is the depth of elastic deflection, and h_{pile} is the pile-up height that can be measured by observing residual indent. Using the term for rough surface effect described previously, the contact depth equation can be expressed as

$$h_c = h_{max} - h_d + h_{pile} - 2.46 \times R_a \quad . \tag{2}$$

The difference between Eqs. (1) and (2) is clearly observed in Fig. 1, which shows the changes in hardness (H) and Young's modulus (E) with average surface roughness. Poisson's ratios of 0.277 for W and 0.315 for Ni (previously determined by ultrasonic technique in

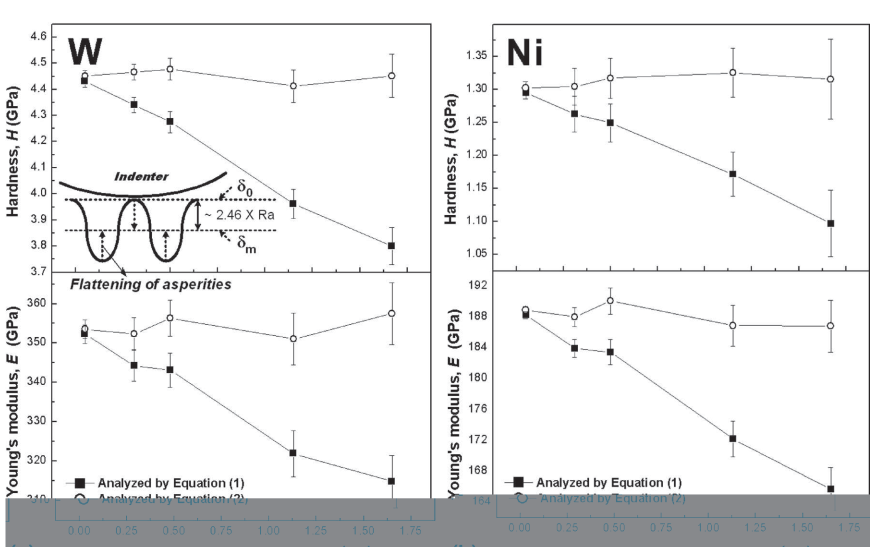

FIG. 1. Variation in hardness, H, and Young's modulus, E, with average surface roughness, R_a: (a) W and (b) Ni.

authors' unpublished work) were used in all the calculations of E from E_r. As shown in the figure, both H and E obtained using contact depth of Eq. (1) decrease with increasing surface roughness. Comparison of the results for R_a ~1.6 μm with those for R_a ~0.1 μm shows that H and E decrease by 14.24% and 10.6% respectively for W, and by 15.32% and 11.99% respectively for Ni. The variations with surface roughness are not easy to explain simply because, for a given indentation load, the real values of H and E in a material must be insensitive to roughness. On the other hand, in Fig. 1, the H and E values according to Eq. (2) do not vary significantly with increase in average surface roughness: the averages and standard deviations of the H and E values from Eq. (2) were 4.45 ± 0.025 GPa and 354.14 ± 2.745 GPa respectively for W, and 1.31 ± 0.01 GPa and 188.14 ± 1.392 GPa respectively for Ni.

Direct measurements of the maximum load (P_{max}) and stiffness (S) at a given indentation depth (h_{max} = 50 μm in this study) can also be useful in understanding the underestimations of the H and E analyzed by Eq. (1). Assuming that H and E are independent of contact depth, the values of P_{max} and S should have a linear relationship with h_c^2 and h_c, respectively. The measured P_{max} and S showed a clear relation with contact depth reformulated by Eq. (2) for each roughness condition, as shown in Fig. 2. In the figure, a larger h_c means a flatter original surface (i.e., a smaller R_a). Note that the quadratic curves for P_{max} versus contact depth (Fig. 2) seem linear because they cover a very local region of the quadratic curve through the origin. The values of maximum load and stiffness decreased with increasing average surface roughness as shown in Fig. 2, while the variations in the contact area (A_c) analyzed by Eq. (1) are negligible; the maximum differences were 0.58% for W and 0.37% for Ni. It can be concluded that the H and E are underestimated with increasing average surface roughness (R_a) since directly measured maximum load and stiffness are decreased while the change in A_c by Eq. (1) is negligible with increasing R_a.

These results indicate that, for a material having surface roughness of R_a, the values of P_{max} and S measured at h_{max} are the same as those measured at (h_{max} −2.46 × R_a) for the material having an ideally flat surface. This result can be simply explained by the shift in the indentation load-depth curve shown in Fig. 3, where representative indentation load-depth curves for W and Ni having the largest and smallest R_a are presented. Note that the zero load index (at which the indentation equipment recognizes initial contact) is set as small as the load resolution to minimize possible artifacts. When the rough-surface curve is shifted by (−2.46 × R_a) along the depth axis, the experimentally measured P_{max} and S are the same as those measured at (h_{max} − 2.46 × R_a) for an ideally flat surface. The area under the loading curve is the energy input into a material by indentation during the loading sequence. With the shift in the load-depth curve, the energy integrated from (−2.46 × R_a) to (h_{max} − 2.46 × R_a) for a rough surface was greater than the energy integrated from zero to (h_{max} − 2.46 × R_a) for a flat surface, as shown in Fig. 3. This additional energy for indentation on a rough surface might be expended in flattening the asperities within A_c.

Collectively, the contact depth model proposed here seems very useful for considering surface roughness effects during instrumented indentation measurement of H and E. While the H and E based on the conventional 'flat surface' assumption significantly decreases with increasing surface roughness, the values analyzed in the proposed rough-surface model are almost insensitive to original surface roughness. If the applicability of this model can be extended to nanoindentation scale, which is not verified here, one may expect that more accurately measuring small-scale mechanical properties of a material having uncontrollable roughness (such as very thin film and bio-tissue) is possible.

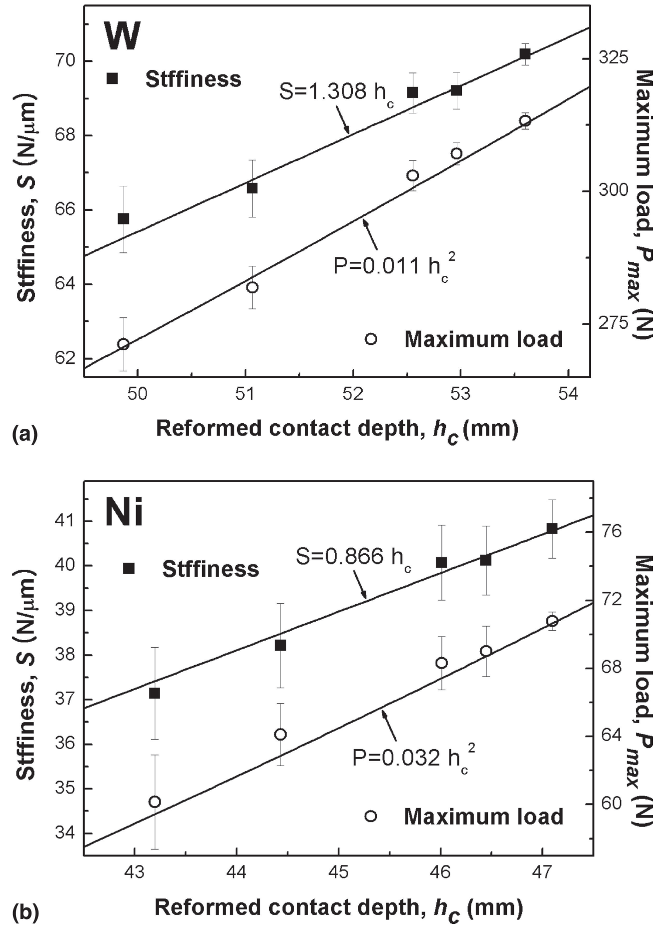

FIG. 2. Dependences of the maximum load, P_{max}, and stiffness, S, on the contact depth, h_c, which was reformed according to the model proposed in this study: (a) W and (b) Ni.

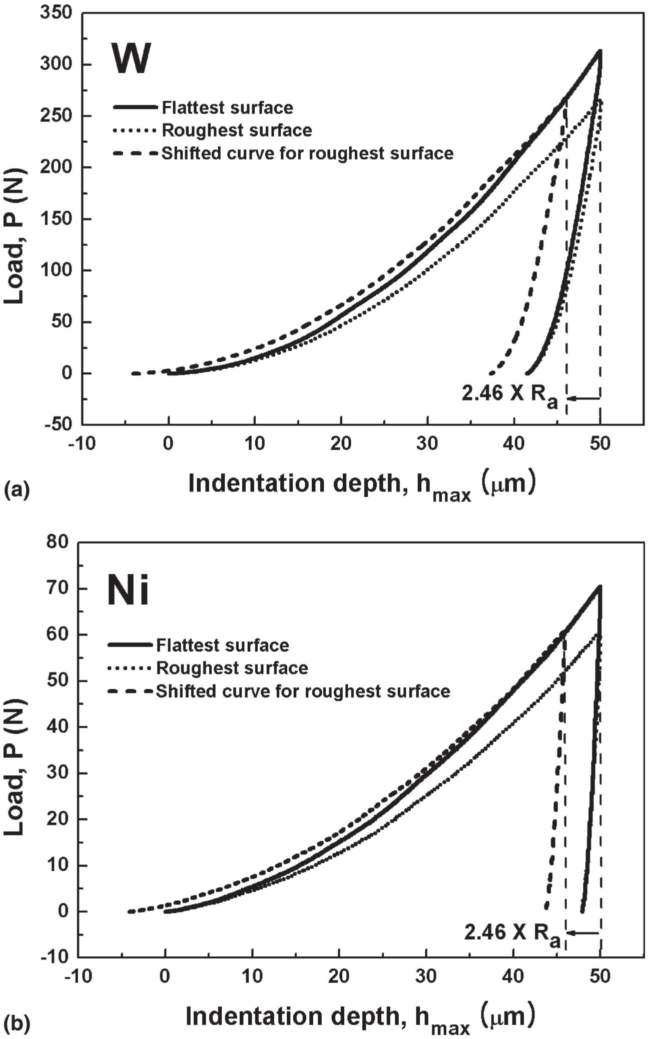

FIG. 3. Indentation load-depth curves obtained from the flattest and the roughest samples of (a) W and (b) Ni. In the case of the roughest sample, the curves shifted by (2.46 × R_a) are shown together for comparison purpose.

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