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FINITE ELEMENT STUDIES OF THE INFLUENCE OF PILE-UP ON THE ANALYSIS OF NANOINDENTATION DATA

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ABSTRACT

Methods currently used for analyzing nanoindentation load-displacement data give good predictions of the contact area in the case of hard materials, but can underestimate the contact area by as much as 40% for soft materials which do not work harden. This underestimation results from the pile-up which forms around the hardness impression and leads to potentially significant errors in the measurement of hardness and elastic modulus. Finite element simulations of conical indentation for a wide range of elastic-plastic materials are presented which define the conditions under which pile-up is significant and determine the magnitude of the errors in hardness and modulus which may occur if pile-up is ignored. It is shown that the materials in which pile-up is not an important factor can be experimentally identified from the ratio of the final depth after unloading to the depth of the indentation at peak load, a parameter which also correlates with the hardness-to-modulus ratio.

INTRODUCTION

This paper deals with effects of pile-up on the measurements of contact area, hardness and effective modulus from nanoindentation load-displacement data using the Oliver-Pharr method [1]. The effects are investigated using finite element simulation of the indentation of an elastic-plastic half-space by a rigid conical indenter with a semi-vertical angle of 70.3°, which gives the same area to depth ratio as the Berkovich pyramidal indenter used frequently in nanoindentation experiments. The Oliver-Pharr method may be applied to the simulated indentation load-displacement data in the following manner. First, the unloading curve is fit to the power-law relation:

$$P = B(h - h_f)^m \tag{1}$$

where P is the load, h is the displacement, h_f is the final displacement after complete unloading, and B and m are power law fitting parameters. Next, the unloading stiffness, S, is found as the derivative of Eqn.1 at the maximum depth of indentation, $h = h_{max}$, using:

$$S = dP/dh(h = h_{max}) = Bm(h_{max} - h_f)^{m-1} \tag{2}$$

The projected contact area, A, hardness, H and effective modulus of the material, E_{eff}, (assuming that the indenter is rigid), are then determined using:

$$A = \pi(h_{max} - 0.75P_{max}/S)^2 \tan^2(\phi) \tag{3}$$

$$H = \frac{P}{A} \quad (4)$$

$$E_{eff} = \frac{E}{1-\nu^2} = \frac{S\sqrt{\pi}}{2\sqrt{A}} \quad (5)$$

where E and ν are the Young's modulus and Poisson's ratio of the material and ϕ is the semi-vertical angle of the cone [1].

The attractiveness of this approach is that no direct measurements of contact areas are needed, thus facilitating the measurement of mechanical properties from very small indentations. Clearly, however, the accuracy of the method depends on how well Eqns.3-5 describe the deformation behavior of the material. In this regard, it is important to note that Eqns.3-5 were obtained from a purely elastic contact solution developed by Sneddon [2], and thus may not work well for elastic/plastic indentation. One reason for this is that in Sneddon's elastic solution, the material around the indenter always sinks in, while for elastic/plastic indentation, there may be either sink-in or pile-up. Therefore, it is not surprising that method has been found to work well for hard materials, as shown in [1], but one may expect errors in the application of the method to softer materials (see [3], for example).

Here, we investigate by finite element methods the influences that pile-up has on the accuracy with which hardness and modulus can be measured by nanoindentation methods. The key parameter in the investigation is the contact area, which may be determined either from application of Eqn.3 to the simulated indentation load-displacement data (the Oliver-Pharr method), or by direct examination of the contact profiles in the finite element mesh. The two areas obtained from the finite element calculations can be substituted into Eqns.4-5 to obtain two separate estimations of the hardness and elastic modulus. If the Oliver-Pharr method outlined in Eqns.1-5 works well, then one should expect good agreement between the area and hardness obtained by the method and by finite element analysis. Also, the estimation of effective modulus obtained by Eqn.5 should match well with the value for effective modulus that was used as input in the finite element program. What will be shown is that the measurement of H and E using Eqns.3-5 works well when there is little pile-up, but when the pile-up is large, significant errors can result.

FINITE ELEMENT PROCEDURES

Elastic/plastic indentation was simulated using the axisymmetric capabilities of the ABAQUS finite element code. The indenter was modeled as a rigid cone with a semi-vertical angle of 70.3°. All simulations were performed to the same depth, h_{max} = 500 nm, using a finite element mesh similar to one used previously [3].

The material examined in the simulation had a Young's modulus E=70 GPa and Poisson's ratio of $\nu = 0.25$. The von Mises yield criterion with isotropic hardening was used to model the material behavior, and no friction was assumed between the punch and material. The yield stress input in the code was varied between σ_y = 114.25 MPa and σ_y = 26.62 GPa to simulate different materials. Two separate cases of strain hardening were considered, one with no strain hardening, that is, a work hardening rate $\eta = d\sigma/d\epsilon = 0$, (i.e., the material was assumed to be elastic-perfectly plastic) and the other with a work hardening rate of

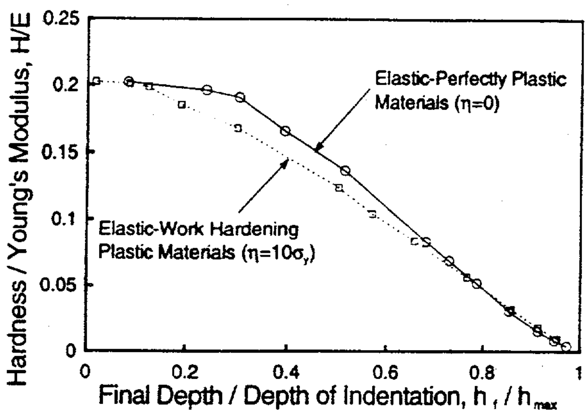

Fig.1. The dependence of H/E on h_f/h_{max} for the two different types of materials under consideration.

$\eta = 10\sigma_y$. These yield strengths and work hardening rates cover a wide range of metals, ceramics and polymers.

Note that in the finite element analysis, the value of the Poisson's ratio was kept constant at $\nu = 0.25$. The influence of the Poisson's ratio on conical indentations in elastic-plastic materials will be reported at a later date (see [6]).

In the course of our finite element study, we found a convenient. experimentally measurable parameter which can be used to identify the expected indentation behavior of a given material. This parameter is the ratio of final depth, h_f, (i.e. depth of the indentation impression after unloading) to the depth of the indentation at peak load, h_{max}. Note that this parameter, h_f/h_{max}, can be easily extracted from the unloading curve in a nanoindentation experiment. Furthermore, because the conical indenter has a self-similar geometry, h_f/h_{max} does not depend on the depth of indentation in a given material. The natural limits for this parameter are $0 \leq h_f/h_{max} \leq 1$. The lower limit corresponds to the fully elastic case, whereas $h_f/h_{max} = 1$ corresponds to the case of no elastic recovery after unloading. In the next section, a parametric study of the dependencies of different measured material properties on h_f/h_{max} will be presented which reveals the importance of this parameter for nanoindentation experiments.

FINITE ELEMENT RESULTS AND DISCUSSION

We start the discussion with Fig.1, which shows the dependence of the hardness to Young's modulus ratio, H/E, on h_f/h_{max}. In computing the data used for the plot, h_f and h_{max} were taken directly from the load-displacement curves generated in the finite element simulations, H was computed as the indentation load divided by the actual projected contact area at peak load determined by examination of the finite element mesh, and the value of E was that used as input for the finite element calculations. The H/E ratio is thus that of the simulated material rather than the value that would be derived from an analysis of the load-displacement data by the Oliver-Pharr method.

An examination of the curves in Fig.1 reveals that:

$$\lim_{h_f/h_{max} \to 1} H/E = 0 \tag{6}$$

$$\lim_{h_f/h_{max} \to 0} H/E = 0.207 \tag{7}$$

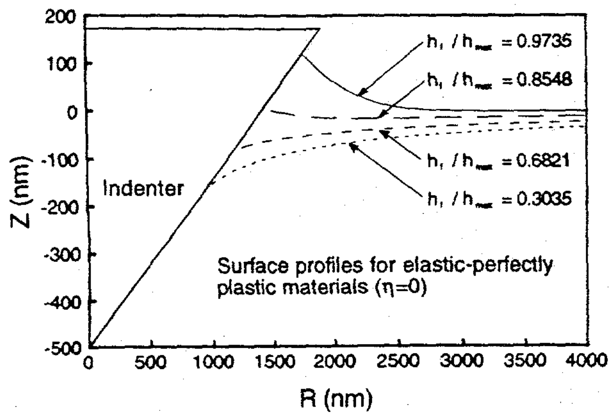

Fig.2. Surface profiles for different values of h_f/h_{max} for elastic-perfectly plastic materials ($\eta = 0$).

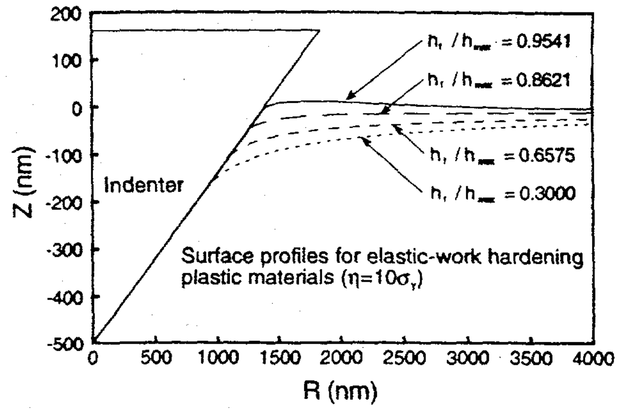

Fig.3. Surface profiles for different values of h_f/h_{max} for elastic-work hardening plastic materials ($\eta = 10\ \sigma_y$).

The first limit represents the case of rigid/plastic deformation, for which there is no elastic recovery during unloading and $h_f = h_{max}$. The second limit (at $h_f/h_{max} = 0$) represents purely elastic behavior. The limiting value $H/E = 0.207$ is a little surprising, since Sneddon's analysis for elastic indentation by a rigid cone gives $H/E = 0.191$. (Note that in deriving this result, it is assumed that the hardness is the indentation load divided by the projected contact area at peak load rather than the projected area of the residual hardness impression.) However, in a separate study [5], we have recently shown that the difference between Sneddon's solution and the finite element result is due to the fact that Sneddon's analysis applies to small deformations while the finite element code accounts for finite deformations. When the effects of finite deformations are included, the finite element calculations give the right limits at both ends of the curves.

Note that if for a given material the ratio h_f/h_{max} is known, then one can make predictions about the value of H/E based on the curves shown in Fig.1. Furthermore if it is assumed that two curves represent extreme cases and that data for most real materials lie between them, then a mid-line can be drawn between the two curves which can be used to estimate the H/E ratio from a known value of h_f/h_{max}. An error analysis showed that the use of such a curve for H/E determination will have less than a 15% error for $0.8 < h_f/h_{max} < 1$ and less than a 7% error for $0.0 < h_f/h_{max} < 0.8$.

Figures 2 and 3 show the surface profiles for the two different types of materials under consideration. Examination of these figures reveals that the amount of pile-up or sink-in depends on the amount of work hardening as well as on the value of h_f/h_{max}. More specifically, the amount of pile-up is large only when h_f/h_{max} is close to one and the amount of work hardening is small. It should also be noticed that for $h_f/h_{max} < 0.7$, very little pile-up is found no matter what the work hardening behavior of the material is.

These observations are particularly important when one considers the results of the contact area estimations shown in Fig.4. The contact area is normalized with respect to the shape function (or area function) of the indenter, that is, the projected area when no pile-up or sink-in occurs. From this figure one may see that since the Oliver-Pharr method is based on an elastic analysis, it fails to predict the correct contact areas for materials in which pile-up is important. Furthermore, when $h_f/h_{max} > 0.7$, the accuracy of the Oliver-Pharr method depends significantly on the amount of work hardening in the material. If

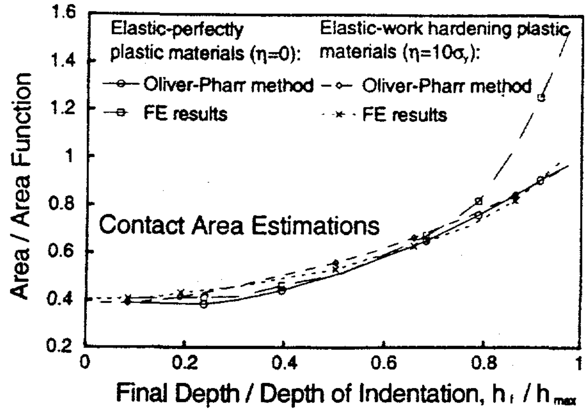

Fig.4. Contact areas as obtained by the Oliver-Pharr method and from finite element calculations.

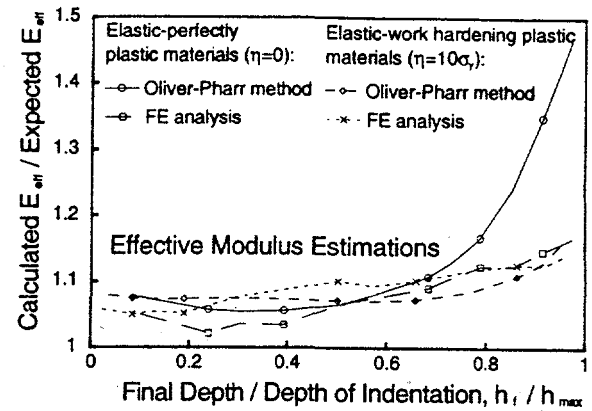

Fig.5. Estimations of effective modulus according to Eqn.5 with contact areas obtained by the Oliver-Pharr method and from finite element calculations.

the material is elastic-perfectly plastic, the Oliver-Pharr method underestimates the contact area by more then 50% at $h_f/h_{max} = 0.9735$. On the other hand, contact areas for materials with a large amount of work hardening are predicted very well by the method. Note that from the experimental point of view, it is not possible to predict if a material work hardens based solely on the load-displacement data. Therefore, in an indentation experiment, care needs to be exercised when $h_f/h_{max} > 0.7$, since use of the Oliver-Pharr method can lead to large errors in the contact area. On the other hand, when the pile-up is small, i.e., $h_f/h_{max} < 0.7$, then, according to Fig.4, the contact areas given by the Oliver-Pharr method match very well with the contact areas obtained from the finite element analysis, independent of the amount of work hardening. Since the hardness of the material is given by $H = P/A$, where P is measured during the experiment, the inaccuracies in the contact area will lead to similar inaccuracies in the hardness (in a reciprocal sence).

The same effects may be seen in effective modulus calculations as shown in Fig.5. The effective modulus was calculated using Eqn.5 with two different estimations for the contact area (the Oliver-Pharr method and the finite element mesh). These calculated effective moduli are normalized with respect to the effective modulus used as input in the finite element simulations (the expected E_{eff}). As in the case of the hardness, the effective modulus will be overestimated when pile-up is significant.

Another noteworthy feature of Fig.5 is that even when the amount of pile-up is negligible, that is, materials with a high yield stresses and/or high work hardening (characterized by h_f/h_{max} <0.7), the Oliver-Pharr method overestimates the effective modulus by 2 to 9%. As shown in Fig.5, this overestimation is predicted even at values of h_f/h_{max} close to 0. In fact the value of effective modulus at $h_f/h_{max} = 0$ is overestimated by 6 to 9%, depending on the method used in calculation. This problem is addressed in [5], in which it is shown that corrections to Sneddon's solution are needed in order to account for finite deformations and rotations that are automatically taken into account in the finite element calculations. The magnitude of the correction depends on the Poisson's ratio, ν, and the cone angle, ϕ. According to [5], the correction will reduce the estimated effective modulus by 8.53%, which is very close to what is needed to correct the moduli deduced from the finite element load-displacement data to the expected value. Thus, for $h_f/h_{max} < 0.7$, there is a simple explanation for the error in the modulus observed in the finite element results.

The same is not true, however, when $h_f/h_{max} > 0.7$. In this range, the effective modulus is overestimated by 10 to 16%, even if correct contact area is used in Eqn.5. This is due to the fact that the plastic properties of the material effect the unloading curves in such a way that the analysis can no longer be done by means of elastic solutions only.

CONCLUSIONS

The results of a finite element study have shown that pile-up can have important consequences for the measurement of contact area, hardness and effective modulus by nanoindentation methods. The parameter h_f/h_{max}, which can be measured experimentally, can be used as an indication of when pile-up may be an important factor. Pile-up is significant only when $h_f/h_{max} > 0.7$ and the material does not appreciably work harden. For such materials, failure to account for the pile-up can lead to an underestimation of the contact area deduced from nanoindentation load-displacement data by as much as 50%. This, in turn, results in an overestimation of the hardness and elastic modulus. When $h_f/h_{max} < 0.7$, or in all materials that strongly work harden, pile-up is not a significant issue, and currently existing nanoindentation data analysis procedures can be expected to give accurate results.

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