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# ANALYSIS OF ELASTIC AND PLASTIC DEFORMATION ASSOCIATED WITH INDENTATION TESTING OF THIN FILMS ON SUBSTRATES

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**Abstract**—A study has been made of the elastic and plastic deformation associated with sub-micrometer indentation of thin films on substrates using the finite element method. The effects of the elastic and plastic properties of both the film and substrate on the hardness of the film/substrate composite are studied by determining the average pressure under the indenter as a function of the indentation depth. Calculations have been made for film/substrate combinations for which the substrate is either harder or softer than the film and for combinations for which the substrate is either stiffer or more compliant than the film. It is found, as expected, that the hardness increases with indentation depth when either the yield strength or the elastic modulus of the substrate is higher than that of the film. Correspondingly, the hardness decreases with indentation depth when the yield strength or elastic modulus of the substrate is lower than that of the film. Functional equations have been developed to predict the hardness variation with depth under these different conditions. Finite element simulation of the unloading portion of the load–displacement curve permits a determination of the elastic compliance of the film/substrate composite as a function of indentation depth. The elastic properties of the film can be separated from those of the substrate using this information. The results are in good agreement with King's analytical treatment of this problem.

## INTRODUCTION

Knowledge of the strengths of metallic thin films bonded to substrates is becoming more and more important due to the increased use of various metallization techniques in integrated circuit devices. This is important because an understanding of the mechanics of deformation permits one to better predict the conditions leading to failure in these thin film structures. This knowledge also permits better design from a structural point of view. The sub-micrometer indentation technique is emerging as one of the popular ways to study the elastic and plastic deformation of thin films on substrates (Pethica *et al.*, 1983; Doerner and Nix, 1986; Doerner *et al.*, 1986). This technique is particularly attractive because it provides information about elastic and plastic deformation on a highly localized scale and because it is simple to use. In the present study we describe the elastic and plastic deformation associated with the indentation of thin films on substrates from a continuum point of view. We have used the finite element simulation technique to study the effects of the yield strength and elastic modulus of the film and substrate on the hardness and elastic compliance of the film/substrate composite.

Although indentation tests have been used for a long time, few theoretical treatments of indentation deformation are available. Such treatments are required to obtain fundamental information about the elastic and plastic properties of the film from an indentation experiment. For an introduction to some of the published literature on this subject, the interested reader may refer to a recent paper (Bhattacharya and Nix, 1988). In this work we concentrate on small indentations of the kind produced in typical sub-micrometer indentation tests used for thin films on substrates. The hardnesses of several thin film materials using sub-micrometer indentation tests have been measured (Doerner and Nix, 1986). The elastic properties of these films were also determined as a function of film thickness and an empirical equation to describe the effect of the substrate on the compliance of the film/substrate composite was suggested. This problem was later examined analytically by King (1987), who developed a theoretical analysis for determining the compliance. While it is possible to study elastic indentation of thin films using analytical methods, elastic–plastic indentations are sufficiently complex that finite element techniques are required. A finite element study of indentation of bulk materials has been conducted recently by the present authors (Bhattacharya and Nix, 1988). We showed that the basic features of the indentation experiment can be described using the finite element technique in conjunction with relatively simple material behavior. In this paper we extend the analysis to the problem of thin films on substrates. Here we analyze the effect of the properties of both the film and the substrate on the hardness and the elastic compliance of the film/substrate composite.

## THE FINITE ELEMENT MODEL

Sub-micrometer indentation testing permits the measurement of force–distance relations on a very small scale; a detailed description of this can be found elsewhere (Pethica *et al.*, 1983; Doerner and Nix, 1986). Simulations of these force–distance relations for the indentation of thin films on substrates using a rigid indenter were performed using the large strain elasto-plastic feature of the ABAQUS (1985) finite element code, with tensile stress–strain data as input. Film and substrate materials with various yield strengths and Young's moduli were studied. The quasi-static nature of the process permits us to use the static analysis performed by the program. Underlying the approach in this code is the discretization of the continuum involved (the layer to be indented here); the indenter was considered to be perfectly rigid. Also, an important feature of this program involves the capability to model contact between the indenter and the sample as a sliding interface. The initial nodal gaps between the indenter and the surface of the specimen were prescribed; the program automatically keeps track of their change and indicates any gap closure or opening in a particular specified direction. These interface elements thus simulate contact between the indenter and the specimen surface. Whenever the closure distance between the indenter and the specimen becomes zero, contact is assumed and an external reaction force is exerted on that particular material point to keep it moving along with the indenter. Because the program calls for incremental loading and also makes use of interface elements, the expanding contact area associated with indentation occurs naturally whenever new interface elements come into contact.

In this analysis, the indenter and specimen are treated as bodies of revolution to avoid the inherent three-dimensional nature of the problem of indentation with a pyramid shaped indenter (Pethica *et al.*, 1983; Doerner and Nix, 1986). This approximation is considered to be acceptable for the case of continuum plasticity; a three-dimensional analysis would be needed to treat crystal plasticity. In the present treatment the pyramid indenter was approximated by an axisymmetric cone (having a perfectly sharp tip) of equal volume for a given indenter depth. In actual test conditions, the indenter tip has a finite radius, thus giving rise to a somewhat different response. The specimen consists of a thin film (1 $\mu$m thick) on a semi-infinite substrate approximated as a plate two orders of magnitude thicker than the film. Perfect contact between the film and the substrate is assumed and both film and substrate were considered to be initially stress free. The indenter and specimen are shown schematically in Fig. 1, along with the appropriate boundary conditions for the problem. Symmetry properties have been used to simplify the boundary conditions. During preliminary simulations the boundary condition on the surface on the right-hand side of the specimen was changed from fixed radial displacements to traction free; this change had no effect on the indentation parameters, thus showing that this boundary was indeed remote. Because very small indentations were being simulated, the meshes near the indenter needed to be very fine to be able to describe the deformation and stress gradients associated with indentation with sufficient accuracy. Thus, extremely fine mesh sizes were used under the indenter; they became progressively coarser at distances further away from the indenter. Axisymmetric four noded elements were used for the continuum. In order to obtain an accurate estimate of the radius of the contact area, an extremely fine mesh thickness of the

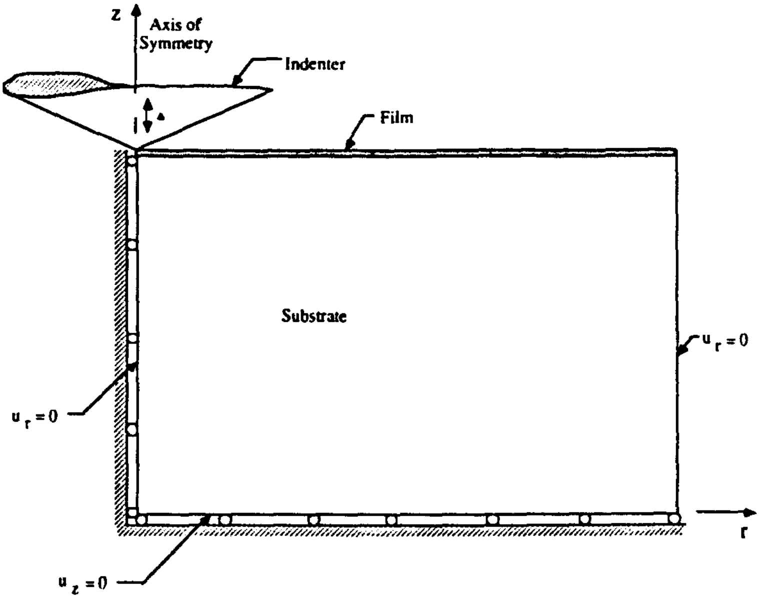


Fig. 1. Schematic diagram of the specimen under the indenter showing the boundary conditions of the indentation problem.

order of 0.02 $\mu$m had to be used along most of the indenter contact surface. To keep the required computer time within limits, a total of 461 elements including the interface elements were used to represent the deformed material. Figure 2 shows a magnified view of the elements near the indenter and the staircase arrangement for the other elements at points further away from the indenter.

To simulate a typical indentation process, a downward displacement (negative $z$-direction in Fig. 1) was imposed on the indenter; this causes the indenter to push into the surface of the material. At the end of the indentation experiment the indenter was given an upward displacement until it was free of contact with the specimen. For a given indenter displacement, the corresponding load was determined by summing the reaction forces at the contact node points on the indenter. The interface between the specimen and the indenter was assumed to be frictionless since no noticeable change in the load–displacement response was observed by using a friction coefficient of 1. The mesh thickness of 0.02 $\mu$m along the indenter contact surface was determined to be acceptable by finding the mesh size below which no further significant changes in the indentation load–displacement response were observed.

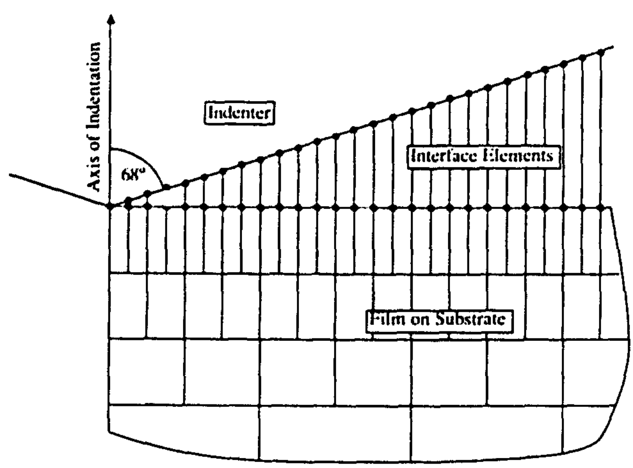


Fig. 2. Detailed pattern of the mesh distribution near the indenter showing the interface elements.

Table 1. Yield strengths and Young's moduli used for the finite element simulation of indentation of thin films on substrates ($\nu_f = \nu_s = 0.278$)

| | | | | | | |
|---|---|---|---|---|---|---|
| Yield strength (MPa) | | | | | | |
| $\sigma_f$ | 485 | 485 | 2070 | 4410 | 2070 | 4410 |
| $\sigma_s$ | 4410 | 2070 | 4410 | 485 | 485 | 2070 |
| $\sigma_f/\sigma_s$ | 0.11 | 0.233 | 0.47 | 9.13 | 4.29 | 2.13 |
| Young's modulus (GPa) | | | | | | |
| $E_f$ | 46.5 | 76 | 127 | 207 | 345 | |
| $E_s$ | 127 | 1237 | 127 | 127 | 127 | |
| $E_f/E_s$ | 0.367 | 0.6 | 1.0 | 1.67 | 2.73 | |

Table 2. Elastic and plastic properties of aluminum and silicon used in the finite element analysis

| Material | Young's modulus (GPa) | Poisson's ratio | Yield strength (MPa) |
|---|---|---|---|
| Aluminum | 75.9 | 0.33 | 485 |
| Silicon | 127 | 0.278 | 4410 |

The constitutive model for the specimen material (both film and substrate) was that of an elastic–plastic von Mises material. Only the case of no strain hardening (i.e. both materials assumed to be elastic–fully plastic) was considered. The elastic and plastic properties of the films and substrates used in the various calculations are given in Tables 1 and 2. The finite element calculations were performed using an IBM 4341 mainframe computer with run times of 1–2 days and also using a Vax II Workstation with run times of 5–6 days for average indentation depths.

## RESULTS AND DISCUSSION

In our previous work (Bhattacharya and Nix, 1988), we showed that the finite element analysis can adequately describe the load displacement response observed in a typical submicrometer indentation test. We also showed how the hardness and Young's modulus can be evaluated from such a simulated result. Figure 3 shows a simulation result for indentation of silicon together with an experimental result reported by Pethica *et al.* (1983). There are some differences in the two results that can be attributed to differences in the actual and assumed material properties and to the discontinuous nature of the simulation procedure.

When an indentation test is performed on a single homogeneous material, the hardness remains essentially constant, irrespective of the depth of indentation. This result is expected

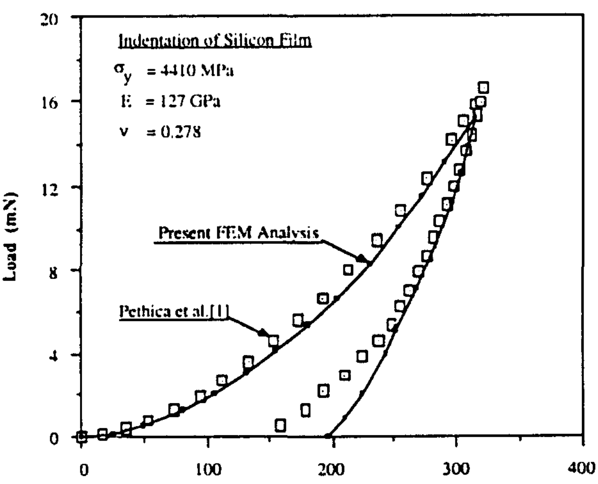


Fig. 3. Comparison of the FEM results of a previous paper (Bhattacharya and Nix, 1988) with the experimental results of Pethica *et al.* (1983) for indentation of silicon.

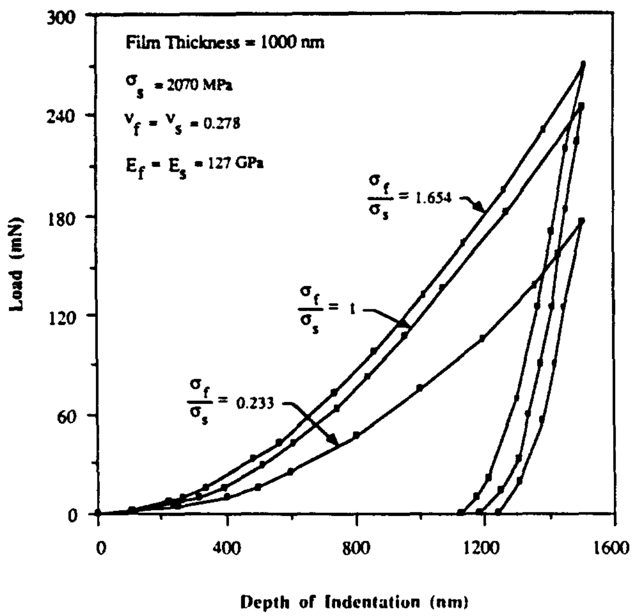


Fig. 4. Effect of relative yield strengths on the load–depth response for thin films on substrates.

in a continuum treatment. Johnson (1970) developed a theoretical expression for hardness using an expanding spherical cavity model of the indentation process. He found that the hardness depends only on the elastic and plastic properties of the material in question and not on the depth of indentation. This result was also found in our finite element study of indentation. In contrast to this behavior, if an indentation is made on a thin film attached to a substrate having different elastic and plastic properties, the hardness associated with the indentation process is expected to continually change due to the gradually increasing influence of the substrate. We next present various results for the indentation of thin films on substrates.

*Hardness*

One of the goals of this work has been to understand how the hardness of a thin film changes with depth of penetration of the indenter. The most important quantities to be considered in this analysis are the thickness of the film and the yield strength, Young's modulus and Poisson's ratio of both the film and the substrate. Strain hardening in the film and substrate could also be considered but it has not been included in the present analysis. In our previous work on the indentation of semi-infinite solids we found that including strain hardening in the analysis does not produce qualitatively significant effects. The response of a material with a high rate of strain hardening is essentially the same as the response of a material with a higher yield strength. In a typical indentation experiment, the elastic–plastic response is characterized by a growing volume of material subjected to a fixed strain rather than by a fixed volume of material subjected to an increasing strain. For this reason it is not especially important to include strain hardening in the analysis.

We consider first the hypothetical cases in which the film and substrate have either different elastic properties (Young's moduli) or different yield strengths. As mentioned above, all simulations have been performed on 1 $\mu$m thick films that are perfectly bonded to a comparatively thick substrate. It is shown below that even though we have used only one film thickness, the formulation developed from these results will also predict the general trends observed for films with different thicknesses. Figure 4 shows the calculated load–displacement response in cases for which the yield strengths of the film and substrate are different but for which both Young's moduli and Poisson's ratio for the film and the substrate are the same. Similarly, Fig. 5 shows the response in cases for which the Young's moduli of the film and substrate are different but for which the yield strengths and Poisson's ratios are the same. From these results, the hardness is calculated as the load divided by

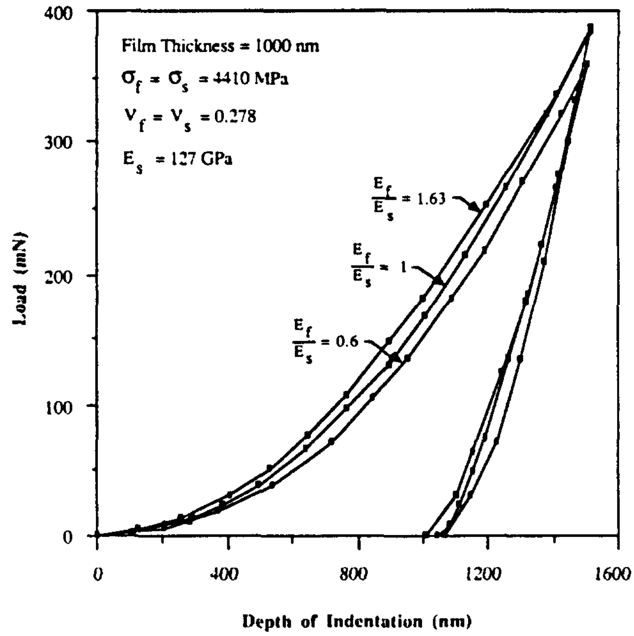


Fig. 5. Effect of relative Young's moduli on the load-depth response for thin films on substrates.

the projected area of contact. These hardness results are shown in Figs 6 and 7. The calculated hardness and the depth of indentation have been non-dimensionalized with the hardness of the substrate and the film thickness, respectively. As intuitively expected, for the case of a softer film on a harder substrate (Fig. 6), the hardness increases with the indentation depth. As seen for this case, the hardness is independent of the substrate for indentation depths less than about 0.3 of the film thickness, after which the hardness slowly increases because of the presence of the substrate. For the case of a harder film on a softer substrate (Fig. 7), the hardness decreases as the depth of indentation increases. In this case, the hardness is constant for indentation depths less than about 0.2 of the film thickness. The regime of constant hardness appears to get smaller as the yield strength of the film increases relative to that for the substrate.

In Figs 8 and 9, we show the hardness results for cases in which the film and substrate have different Young's moduli. It is observed that the variation of hardness with depth of

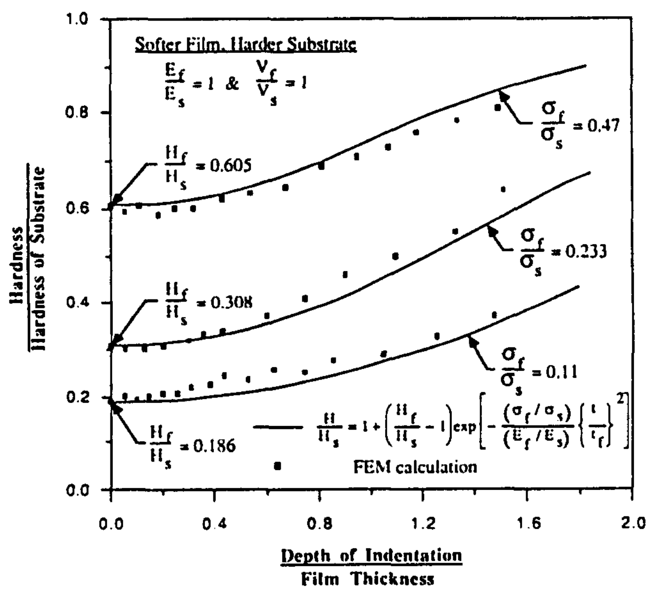


Fig. 6. Effect of relative yield strengths on the hardness of soft films on hard substrates.

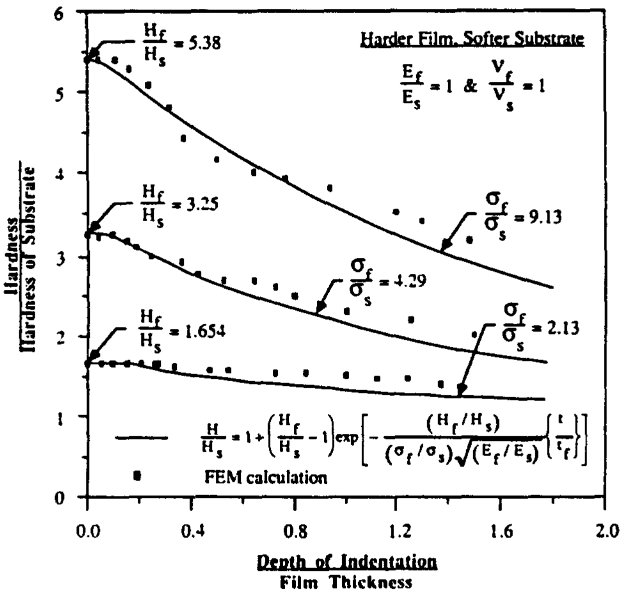


Fig. 7. Effect of relative yield strengths on the hardness of hard films on soft substrates.

indentation in these cases is qualitatively similar to cases in which the film and substrate have different yield strengths, although the hardness changes more gradually than in the previous cases.

The results presented in Figs 6–9 can be used to develop general relations for the hardness variation with indentation depth. It is believed that the hardness results will depend only weakly on Poisson's ratio. For this reason, the effect of Poisson's ratio has not been considered in our analysis. After analyzing these results and fitting with various forms of equations, we have developed two empirical equations which satisfactorily describe the variation of hardness with depth. For the case of a soft film on a harder substrate, the effect of the substrate on film hardness can be described as

$$\frac{H}{H_s} = 1 + \left(\frac{H_f}{H_s} - 1\right)\exp\left[-\frac{(\sigma_f/\sigma_s)}{(E_f/E_s)}\left\{\frac{t}{t_f}\right\}^2\right] \quad (1)$$

where, $E_f$, $E_s$ are the Young's moduli, $\sigma_f$, $\sigma_s$ the yield strengths and $H_f$, $H_s$ the hardnesses

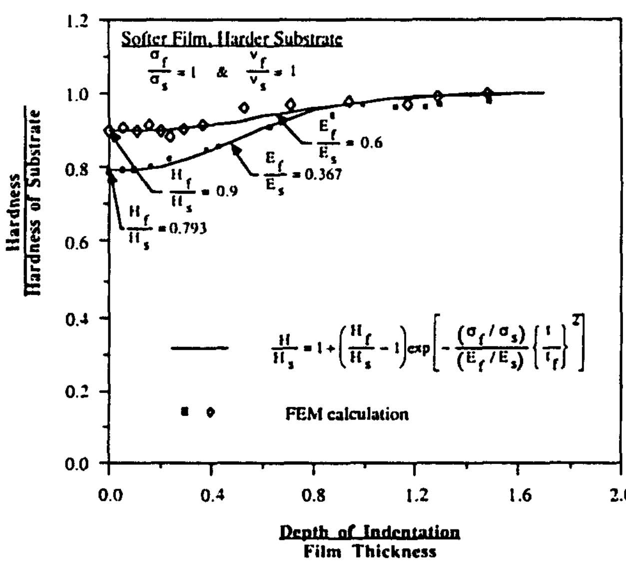


Fig. 8. Effect of relative Young's moduli on the hardness of soft films on hard substrates.

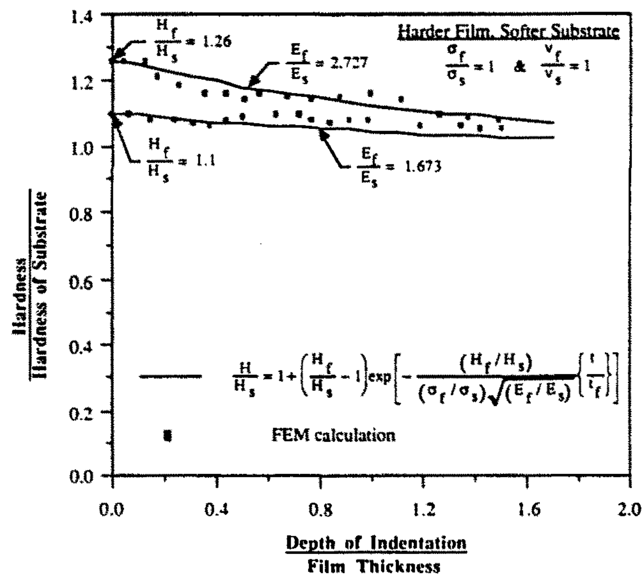


Fig. 9. Effect of relative Young's moduli on the hardness of hard films on soft substrates.

of the film and substrate, respectively. Similarly for the case of a hard film on a softer substrate, the hardness can be expressed as

$$\frac{H}{H_s} = 1 + \left(\frac{H_f}{H_s} - 1\right)\exp\left[-\frac{(H_f/H_s)}{(\sigma_f/\sigma_s)\sqrt{(E_f/E_s)}}\left\{\frac{t}{t_f}\right\}\right]. \tag{2}$$

These two equations are fully nondimensionalized and thus can be used for any film thickness involving any material combination of the film and substrate.

We next discuss the predictive capabilities of these functional equations to describe the hardness of an aluminum film on a silicon substrate and a silicon film on an aluminum substrate. Figure 10 compares the load-displacement response for the indentation of an aluminum film on a silicon substrate with that for a silicon film on an aluminum substrate. The elastic and plastic properties for these materials used in the FEM calculation are given in Table 2. In Fig. 10, we also show the indentation curves for bulk aluminum and silicon. At very small depths of indentation, the load-depth response for the film/substrate

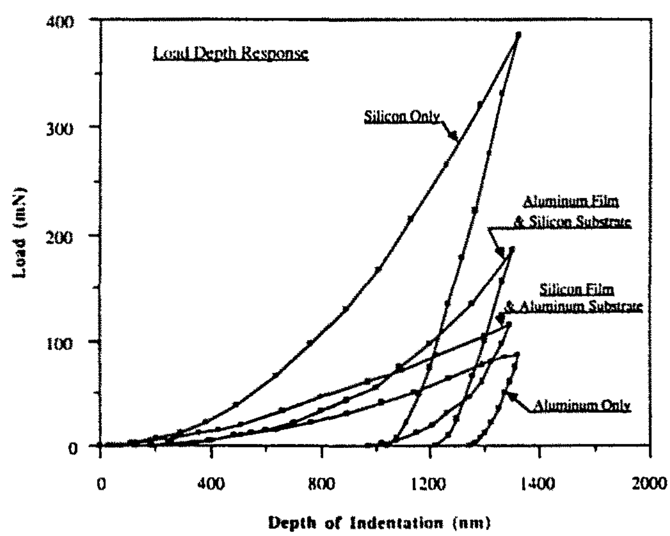


Fig. 10. Load-depth responses for the indentation of an aluminum film on a silicon substrate and a silicon film on an aluminum substrate.

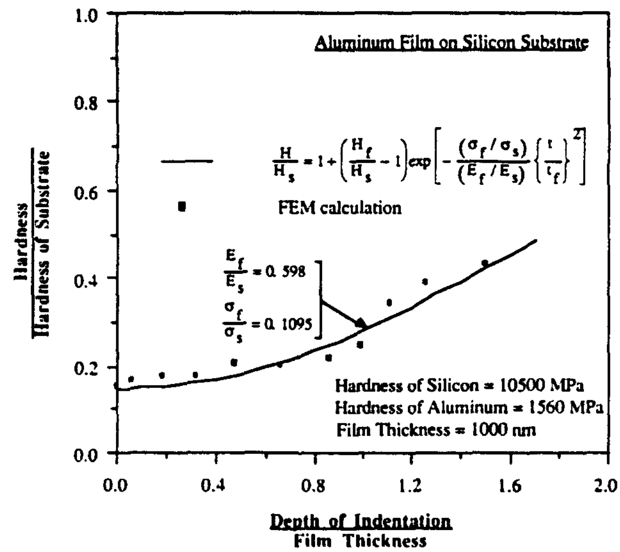


Fig. 11. Comparison of hardness values predicted by eqn (1) with FEM calculations for the case of an aluminum film on a silicon substrate.

composite is the same as the response for the thin film material in bulk form. This result is as expected since for very small indentations only the properties of the thin film are involved. Figure 11 shows the hardness values obtained from Fig. 10 plotted as a function of indentation depth for the case of an aluminum film on a silicon substrate. Similarly, hardness values for a silicon film on an aluminum substrate are plotted in Fig. 12. We also show predictions of eqns (1) and (2), respectively, in these figures. We find that the empirical relations developed for hypothetical films and substrates give a good description for the specific cases of films and substrates composed of aluminum and silicon.

For the case of a soft film on a harder substrate, eqn (1) indicates that as the film is made thinner, it becomes progressively harder. On the other hand, in the case of a harder film on a softer substrate, eqn (2) predicts that as the film thickness is reduced, the hardness of the film decreases. These results suggest that the substrate can play a dominating role in increasing or decreasing the hardness of a film.

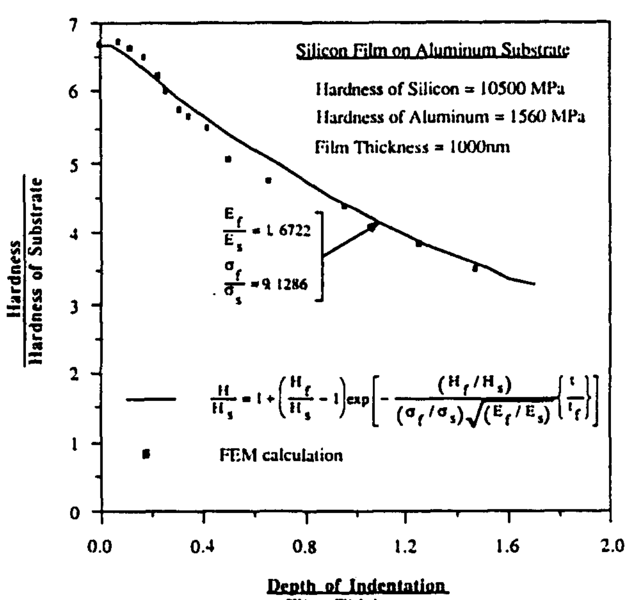


Fig. 12. Comparison of hardness values predicted by eqn (2) with FEM calculations for the case of a silicon film on an aluminum substrate.

*Elastic response*

Recent experimental studies (Pethica *et al.*, 1983; Doerner and Nix, 1986), have shown that in an indentation experiment, the process of unloading is elastic. This is indicated by the linearity of the initial portion of the unloading curve. This has been confirmed by the present authors (Bhattacharya and Nix, 1988), through an FEM analysis of the indentation of silicon and aluminum. We also observed that during the earliest stage of unloading, the contact area under the indenter remains constant. When these conditions of unloading exist, Young's modulus of the material being indented can be determined from the compliance of the film and the plastic depth (Pethica *et al.*, 1983; Doerner and Nix, 1986). As demonstrated previously (Doerner and Nix, 1986), the plot of compliance vs reciprocal plastic depth is linear for a bulk material. However, in the case of thin films on substrates, this linear relationship no longer holds due to the varying contribution of the film and substrate to the measured compliance. There are two limiting cases. For indentation depths that are very small compared to the film thickness, the compliance approaches the value expected for the thin film material whereas for large depths of indentation, it approaches the value expected for the substrate material. The variation of overall compliance of a thin film/substrate composite was studied previously (Doerner and Nix, 1986). Later King (1987) used basis functions and an integral equation technique to perform elastic analyses for punches of different shapes and derived more accurate equations for the compliance in terms of the projected area of contact under the indenter. King's equation, when simplified for an infinitely rigid indenter, becomes

$$\frac{\mathrm{d}h}{\mathrm{d}P} = \frac{1}{\beta\sqrt{A}}\left[\frac{(1-\nu_f^2)}{E_f}\left\{1-\exp\left(-\frac{\alpha' t_f}{\sqrt{A}}\right)\right\}+\frac{(1-\nu_s^2)}{E_s}\left\{\exp\left(-\frac{\alpha' t_f}{\sqrt{A}}\right)\right\}\right] \tag{3}$$

where $A$ is the projected area of contact under the indenter, $\beta$ a numerical factor related to the shape of the indenter and $\alpha'$ a parameter dependent on the depth and is obtainable from a set of curves given by King (1987).

In eqn (3), the value of $A$ is related to the plastic depth through the indenter geometry and hence, $A = k^2h_p^2$, where $k^2 = 24.5$ is a constant related to the geometry of the indenter. With this transformation and normalizing in terms of $t_f/h_p$, we can rewrite eqn (3) as

$$t_f\frac{\mathrm{d}h}{\mathrm{d}P} = \frac{1}{\beta k}\left(\frac{t_f}{h_p}\right)\left[\frac{(1-\nu_f^2)}{E_f}\left\{1-\exp\left(-\alpha''\frac{t_f}{h_p}\right)\right\}+\frac{(1-\nu_s^2)}{E_s}\left\{\exp\left(-\alpha''\frac{t_f}{h_p}\right)\right\}\right] \tag{4}$$

where $\alpha'' = \alpha'/k$.

This equation is expected to be valid for any film thickness $t_f$. Compliances $\mathrm{d}h/\mathrm{d}P$ were directly obtained from the unloading part of the FEM calculations based on a curve fitting procedure described in our earlier analysis (Bhattacharya and Nix, 1988). These compliances were determined for different combinations of indentation depths and film thickness. In Fig. 13, we have plotted $t_f\mathrm{d}h/\mathrm{d}P$ values for film/substrate composites having various Young's moduli ratios and compared them with the predictions of eqn (4). In this treatment the film and the substrate are considered to have the same Poisson's ratio. Calculated values from eqn (4) are seen to be in reasonable agreement with the FEM results for large depths of indentation. But, for smaller depths of indentation, it appears that the values predicted by this equation asymptotically approach the compliance of the film too slowly.

We next consider the elastic response of two specific film/substrate composites, e.g. an aluminum film on a silicon substrate and a silicon film on an aluminum substrate. These results, based on the material properties given in Table 2, are presented in Figs 14 and 15. In these plots, comparison has been made between the FEM calculations and the predictions of eqn (4). The two results are in reasonably good agreement.

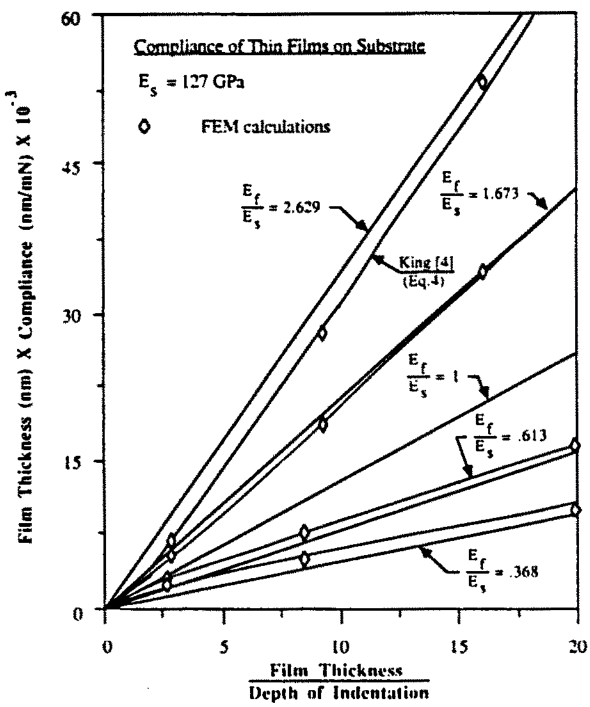


Fig. 13. Comparison of the compliances predicted by eqns (3) and (4) with the FEM calculations for various Young's moduli.

## CONCLUSIONS

The results obtained in this study indicate that the finite element technique can be used to simulate the elastic and plastic response of a sub-micrometer indentation test for thin films on substrates. Based on calculations for two different classes of film/substrate composites, namely soft films on harder substrates and hard films on softer substrates, functional equations have been developed to describe the variation of hardness with depth of indentation. These equations correctly indicate that the film strength increases or decreases as the film thickness is reduced depending on whether the film is softer or harder, respectively,

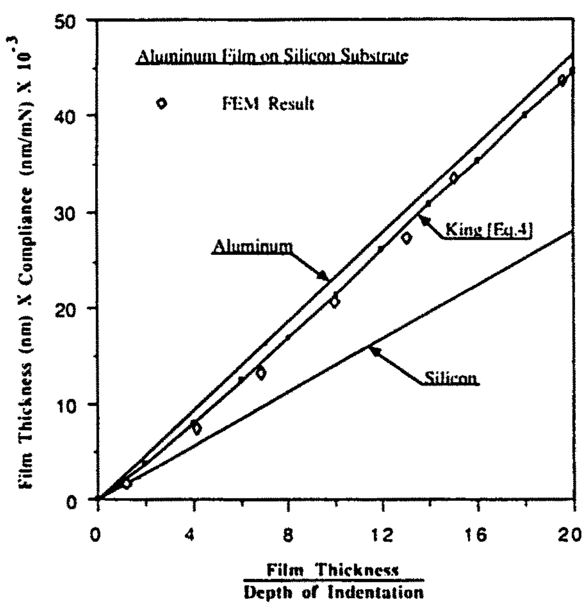


Fig. 14. Comparison of the FEM calculations with the predictions of eqn (4) for an aluminum film on a silicon substrate.

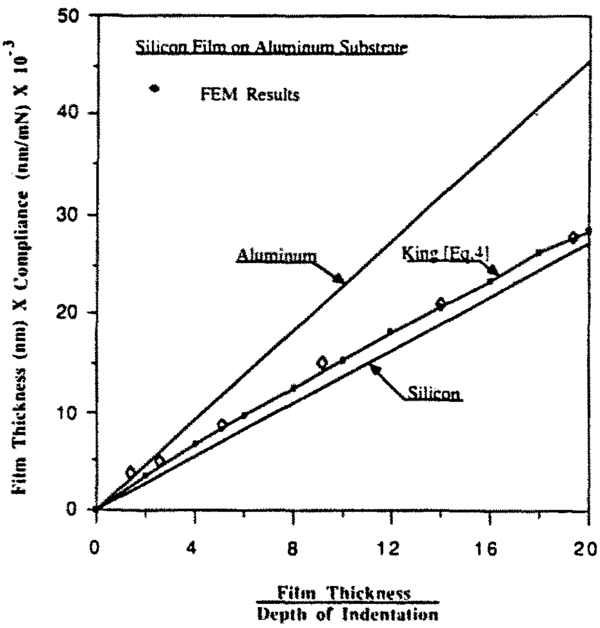


Fig. 15. Comparison of the FEM calculations with the predictions of eqn (4) for a silicon film on an aluminum substrate.

than the substrate. The effect of changing film thickness is found to be larger for the case of a soft film on a harder substrate. It is also shown that the elastic compliance of the film/substrate composite can be determined as a function of indentation depth from the unloading part of the load displacement curves obtained from the FEM analyses. The results are in close agreement with King's analytical treatment of this problem.

Taken altogether, the results of this work provide a scheme by which the hardness of a very thin film can be determined from the measured hardness of a thin film/substrate composite. First the hardness and elastic modulus of the bare substrate would be measured by indentation methods. Then, the hardness and elastic compliance of the thin film/substrate composite would be measured as a function of indentation depth. The variation of hardness with depth would indicate whether eqn (1) or eqn (2) should be used to describe the properties of the composite. The compliance data would be used in conjunction with eqn (4) to determine the elastic modulus of the film or, equivalently, $E_f/E_s$. Finally, eqn (1) or eqn (2), as appropriate, would be inverted to obtain the hardness of the film from the hardness of the thin film/substrate composite.

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