FEM Applied to Evaluate Composite Hardness of SiO₂ Film/316 LSS Substrate System

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Abstract. Due to non-ideal tip, performance of instrument and substrate effect, it is difficult to evaluate either composite mechanical properties of thin film/substrate system or the pure film’s properties. In order to get composite hardness of thin film and how the substrate takes effect of the film’s properties, FEM is used to simulate the indentation process of SiO₂ thin films on 316LSS. With experiments, we observe that the hardness heavily depends the thickness of SiO₂ film with different thickness deposited on 316 LSS by PVD. By FEM simulation and calculation, composite hardness is found to decrease greatly with increasing indentation depth. As thickness of film keeps constant, there exists a critical indentation depth about 1/20 of film thickness, less than which composite hardness may be regarded as film hardness. Discussion indicates FEM analysis method in this paper may not only play role in determining composite hardness of thin film/hardness system, but also provide a method to calculate pure hardness of thin film.

1. Introduction

Depth-sensing indentation is an important technique developed in the past decade with a main purpose to determine the hardness of a solid. However, the method applied to a film deposited on a substrate makes the interpretation of the data complicated because of deformation of the substrate [1], and so it is difficult to attain the true hardness based on the measured load-displacement data of the layered composite structure. The conventional rule of thumb suggests the indentation depth smaller than 1/10 of the film thickness can obtain acceptable result, however, experimental observations suggest that this way will take much error for a hard film/soft system [2]. A number of experimental techniques and models of analysis have been developed with the purpose of extracting the true film hardness from the load-displacement data of a film-on-substrate structure [3-7]. For the case of very thin hard film/soft substrate, it will be more difficult to characterize the hardness either of film or film/substrate system, due to non-ideal tip at shallow indentation and plastic deformation occurs in deep indentation.

Experiment results of thin SiO₂ films deposited on 316LSS, shown in figure 1 (a) and (b), illustrate the dilemma in interpreting indentation data of thin hard film/soft substrate. Figure 1 (a) is the hardness calculated directly by Oliver’s method, revealing the hardness heavily depends on indentation depth. And Figure 1 (b) plots relationship between the hardness and film thickness for a series of SiO₂ films deposited on 316LSS by controlling deposition time, revealing that the hardness varies greatly with the film thickness. Therefore, it makes us impossible to give a correct hardness characterization for such a hard film/soft substrate system. In order to investigate substrate’s deformation on the composite hardness and try to get pure hardness of the film, FEM is applied to
simulate the indentation process and the stress variation during indentation in this paper. By finite element analysis, data such as composite hardness, stress, and indentation depth may be obtained and compared with the experimental results. At very shallow depth and when indentation depth is less that 5% of film thickness, the composite hardness is approximate to hardness of the pure film due to no plastic deformation in the substrate on such occasion. More discussion brought forward an idea to compute mechanical properties of film by FEM.

![Graph](image)

**Figure 1.** Illustrated effects of non-ideal tip and substrate on hardness of SiO$_2$ film/316LSS. (a) Hardness vs. contact depth, (b) Hardness of SiO$_2$ films with different thickness.

2. Mathematical models

The hardness is defined by load over the projected contact area and young’s modulus by functions of stiffness at the initial point of the unloading curve in Oliver&Pharr’s model. Load-displacement curve of a nanoindentation test is illustrated in figure 2, and the loading curve segment of the load-displacement may be characterized by following formulas [1,3]

\[ P = C h^2 \]  
\[ P = K (h - h_r)^m \]

Where \( K \) and \( m \) are the material constant, \( h_r \) is residual indentation depth. At the initial point of the unloading curve, the stiffness \( S \) is expressed as following equation:

\[ \frac{1}{S} = \frac{\Delta h}{\Delta P} \bigg|_{P = P_0} = \frac{1}{\frac{2}{\sqrt{\pi}} E_r \sqrt{A_c} \beta} \]  

With above equation, Young’s modulus of the film \( E_f \) may be determined by following equation.

\[ \frac{1}{E_r} = \frac{1 - \nu_f^2}{E_i} \]  

Where \( E_r \), \( \nu_f \), \( H_f \) are Young’s modulus, Poisson ratio and hardness of the film respectively, and \( E_i \), \( \nu_i \) are Young’s modulus, Poisson ratio of the indenter, and \( E_r \) reduced modulus. Meanwhile, for ideal Berkovich tip, the contact projected depth is calculated by following function:

\[ h_c = h_0 - 0.75 \frac{P_0}{S} \]

Where \( P_0 \) is the maximum load, \( h_0 \) is the maximum dispalcement. Projected contact area \( A_c \) is a function of contact depth:

\[ A_c = 24.5 h_c^2 \]  

The hardness of bulk material defined by the load over projected contact area is
The equations above are suitable for bulk materials. As they are applied to calculate the mechanical properties of film/substrate system, there inevitably exists effect of substrate and tip. If the tip is an ideal one, then the method can give correct composite properties for film/substrate system.

\[ H = \frac{P_0}{A_c} \]  

Figure 2. Load-displacement curve of a nanoindentation.

3. FEM analysis process

Commercial software ANSYS8.0 Multi-physics Package is selected to simulate indentation process. The 3D problem was simplified as an equivalent axisymmetric planar model, as illustrated in figure 3, where angle is 70.3°[8]. The planar model will shorten the analysis time. The diamond tip, whose Young’s modulus \( E = 1140 \text{Gpa} \), Poisson ratio \( \nu = 0.07 \) and friction coefficient=0.2, is modelled as a cone. The measured material is set a cylinder with diameter 10 \( \mu \text{m} \) and the whole thickness of film and substrate is set as concrete 10 \( \mu \text{m} \). \( E = 163 \text{Gpa}, \nu = 0.27, \) coefficient= 0.2, and yield strength \( \sigma = 1.5 \text{GPa} \) are properties of SiO\(_2\) in FEM modelling. Stress-strain relationship of 316LSS was measured by tensile machine Instron1195, as shown in figure 4.

Tip and specimen were meshed by PLANE82, as illustrated in figure 5. Interaction of indenter tip and materials was considered as a contact pair, for which element TARGE169 is applied to the tip and CONTA175 to the specimen. Friction coefficient between the contact pair is set as 0.2. Two steps were conducted to simulate the loading and unloading process, of which the first step increases load from 0 to the maximum value, and the other decreases load from maximum to 0. Each step includes 40 sub-steps. After FEM analysis, important information related to the loading and unloading process was obtained by post process, e.g. the stress, plastic deformation of film and substrate in figure 6. More appropriate post processing makes us plot the load-displacement of a simulated indentation test.

Figure 3. Illustration of Indentation mode. 

Figure 4. Stress-strain relationship of 316LSS.
4. Results and discussion

The FEM results have quite good repeatability in analyzing the film/substrate system. As shown in figure 7 (a), the loading curves with different maximum loads increase with same function. Comparing with the experimental curve figure 7 (b), the displacement of FEM is obvious bigger than the tested one at the same load. Tip’s effect in real application is main reason of the difference. FEM analysis in this paper using ideal tip, theoretically give a correction of composite hardness of film/substrate system.

The FEM results reveal the strong effect of substrate, as shown in figure 7 (c) and (d). Even at indentation depth of 1/10 film the composite hardness is about 5.1 GPa, having much error to the true value about 10.3 GPa. Plastic deformation of substrate leads to a lower composite hardness at very shallow indentation depth. As shown in figure 7 (c), plastic deformation of the substrate begins at about 1/20 film thickness. The composite hardness is approximate to the film hardness when the indentation is less than 1/20 film thickness as shown in figure 7 (c), and the substrate influence was found to be much more severe than what was assumed by the rule-of-thumb. Therefore in order to obtain the film hardness with little substrate influence, indentation experiments should be conducted less than 5% of the film thickness or even smaller. When indentation depth becomes deeper, the maximum stress will be larger and composite hardness will be smaller, and eventually tends to hardness of the substrate 1.95 GPa, as shown in figure 7 (c) and (d). To take pure film thickness, sharper tip, thicker film, and critical indentation depth are strongly recommended for the indentation.
Comparing figure 1(a) and figure 7(d), we may notice that the hardness either by experiment or by FEM tends to the same value when the indentation depth is greater, e.g. half of film thickness, due to the slighter effect of tip. Assuming hardness vs. indentation depth curve (H-D curve) of experiment is as the same as that of the FEM in deeper indentation depth, we propose a method to derive the true mechanical properties of film by series of FEM analysis by following procedures: (1) Assume strain-stress relationship of film and set it as materials for FEM analysis; (2) solve the FEM problem and compare analyzed the H-D curve with the tested one; (c) adjust the strain-stress relationship of film according to the comparing result and perform the FEM again; (4) and so on till the FEM result is very near to the experimental one and then regard mechanical properties of film under such occasion as correct characterization.

5. Conclusion
In this paper, FEM was applied to simulate the indentation process of SiO\textsubscript{2}/316LSS, and effect of substrate during indentation was analyzed. Assuming an ideal tip in simulation and calculation FEM method may give correct characterization of composite hardness of the hard/soft substrate system. For SiO\textsubscript{2}/316LSS system, as thickness of the film keeps constant, there exists a critical indentation depth about 1/20 of film thickness, less than which the composite hardness may be regarded as pure film hardness. Further discussion illuminates us that series of FEM of the hard film/soft substrate system by adjusting mechanical properties of film and comparing simulated result with the experiments is a potential method to calculate pure hardness of thin film.

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