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Study of The Mechanical Properties of Multilayered Coatings at Nanoscale

奈米尺度之多層薄膜的機械性質之研究

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Abstract

In this paper, the mechanical properties of multilayered coatings at nanoscale are studied by nanoindentation and numerical simulation. At first, the thin film Ti, Cu or Cr on substrate Si (i.e., Ti/Si, Cu/Si and Cr/Si) were conducted with nanoindentation, the Young's modulus of Ti, Cu and Cr films can be obtained by using the Oliver and Pharr method. Moreover, based on elastic and elastic-perfectly plastic material models, the finite element method was employed to determine the yield stress of all thin films. In addition, two different multilayered coatings (i.e., Ti/Cu/Si and Ti/Cr/Si) were conducted by nanoindentation and simulated by finite element analysis to assess the effect of various layer of the multilayered coatings. Finally, it is concluded that the maximum depth increases with the increasing of softer middle layer thickness and decreases with the increasing of harder middle layer thickness. The maximum depth decrease either decreasing middle thickness Cu or increasing surface thickness Ti at the multilayered coatings Ti/Cu/Si.

Key Words: Nanoindentation, FEM, thin film, multilayered coatings.

摘 要

本文利用奈米壓痕實驗及數值分析來研究在奈米尺度下的多層薄膜之機械性質，文中首先將單一薄膜為鈦(Ti),銅(Cu)或鉻(Cr)，基材為矽(Si)之試

片，分別利用奈米壓痕實驗，再使用奧利佛和法爾法(Oliver and Pharr method)求出各個薄膜之楊氏係數(Young’s modulus)。其次，本文假設各個薄膜為彈性或彈塑性材料，再利用有限元素法求得各薄膜之降伏強度(yield stress)。對於多層薄膜，本文首先利用奈米壓痕實驗和藉由單一薄膜求得之楊氏係數和降伏強度再利用數值分析，求得多層薄膜當各層膜為不同材料時之影響。結果得到，相對於表層較軟的中層薄膜，當厚度增加愈大壓痕深度愈大，反之，相對表層較硬的中層薄膜，厚度增加愈大壓痕深度愈小。而對於鈦/銅/矽(Ti/Cu/Si)薄膜而言，減小中層薄膜銅的厚度或增加表層鈦薄膜的厚度都會使得壓痕深度變小。

關鍵詞：奈米壓痕、有限元素法、薄膜、多層薄膜

1. Introduction

Multilayered coatings combined the benefit of different material films leading to a further refinement of coating properties. It has attracted attention in both scientific and commercial research in electronics, optical devices and tribological protection applications for many years because it can combine two or more different components for a special purpose. Wen et al. [1] used X-ray diffraction, transmission electron microscopy, and nanoindentation to investigate the microstructure, hardness and elastic modulus of Ag/Co multilayers. The results showed that all the multilayers have well compositionally modulation structure. Chen et al. [2] used X-ray diffraction, scanning electron microscopy, typical secondary ion mass spectroanalyzer, nanoindentation, and scratch test to study the microstructure and mechanical properties of gradient multilayer PVD coatings.

Finite element analysis has been proved as a powerful tool in the nanoindentation simulation. Lichinchi et al. [3] used FEM to simulate nanoindentation of TiN film on HSS by using a commercial finite element code ABAQUS, and both axisymmetric and three-dimensional models were employed. They studied the effect of substrate on the hardness measurement and concluded that both models are similar and provide good fit.

In this paper, the various layer effects of multilayered coatings on nanoindentation with Berkovich probe tip was investigated with the finite element analysis.

2. Nanoindentation

The bulk material as studied with nanoindentation test which is a nanoindenter (Triboscope, Hysitron) assembled on an atomic force microscope (Autoprobe, CP-Research), and a three side pyramidal Berkovich probe tip is used in the test. Five indentations were made in each sample at the same maximum loads. Nanoindentation is performed under a precisely continuous measurement of the load and the depth during the test. Fig. 1 shows a schematic draw of an indentation load vs. depth curve.

In the Oliver and Pharr [4] method, the hardness H and the reduced modulus E_r are derived from

$$H = \frac{P_{max}}{A} \qquad (1)$$

and

$$\left(\frac{dP}{dh}\right)_{unload} = S = 2\beta E_r \sqrt{\frac{A}{\pi}} \qquad (2)$$

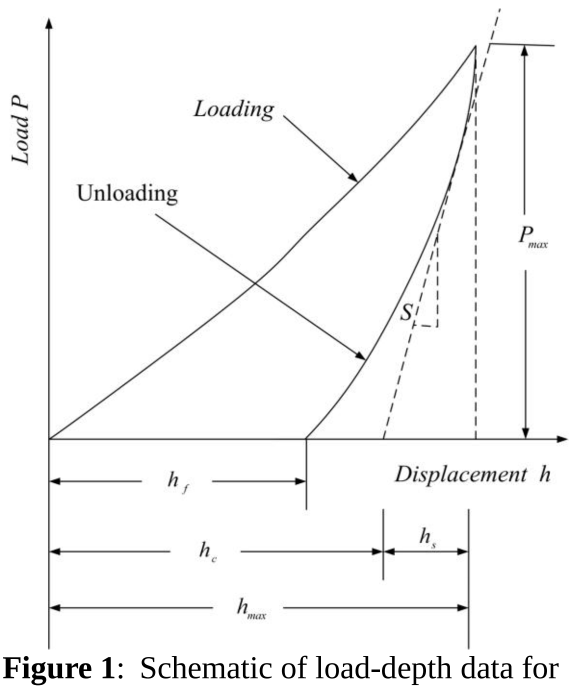

Figure 1: Schematic of load-depth data for a nanoindentation experiment.

where P_{max} is the maximum indentation load, A is the projected contact area, S is the unloading stiffness measured at maximum depth of penetration h, β is a constant that depends on the geometry of the indenter for Berkovich indenter $\beta = 1.034$. The reduced modulus is used in the analysis to take into account that elastic deformation occurs in both the indenter and the specimen, it is given by

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \qquad (3)$$

where E, E_i and ν ν_i are the elastic modulus and Poisson's ratio of the indenter and the specimen material, respectively. For evaluating the elastic modulus E_r, the slope $\left(\frac{dP}{dh}\right)_{unload}$ and the contact area A should be determined precisely. A least mean square fit to 90% of the unloading curve is made according to the hypothesis that the unloading data will be expressed by a power law

$$P = P_{max}\left(\frac{h - h_f}{h_{max} - h_f}\right)^m \qquad (4)$$

For an indenter with a known geometry, the projected contact area is a function of the contact depth. The area function for a perfect Berkovich indenter is given by

$$A = f(h_c) = 24.5\,h_c^2 \qquad (5)$$

Indenters used in practical nanoindentation testing are not ideally sharp. Therefore, tip geometry calibration or area function calibration is needed.

At first the thin films Ti, Cr and Cu were deposited on substrate Si (i.e., Ti/Si, Cr/Si and Cu/Si) by electron beam evaporation at 300nm thickness. The maximum depth vs. Young's modulus curves evaluated from the load-depth data of nanoindentation are plotted in Fig. 2. Moreover, the Oliver-Pharr method is used to calculate the Young's modulus. It shows that the bulk material Si, the curve of Young's modulus vs. maximum depth is like a straight line and the Young's modulus is E_{Si} =165 GPa. For the Cr thin films deposited on Si substrate (Cr/Si), the Young's modulus decreases with maximum depth increasing and when the thin film is Ti/Si or Cu/Si the Young's modulus increases with maximum depth increasing. But the Young's modulus of thin films (Ti, Cr or Cu) is getting close to the straight line of bulk material Si while maximum depth increasing. It is caused by the effect of substrate. In order to avoid substrate effects, the penetration depth is chosen not to exceed 20 % of the film thickness. The Young's modulus of thin films (Ti ,Cu and Cr) on the substrate Si can be found E_{Ti} =134GPa, E_{Cr} =240GPa, and E_{Cu} =110GPa. For determining the yield stress or other material properties, the finite element method based on the elastic-perfectly plastic material model is used to reproduce the load-depth curve. Then, the yield stress can be obtained.

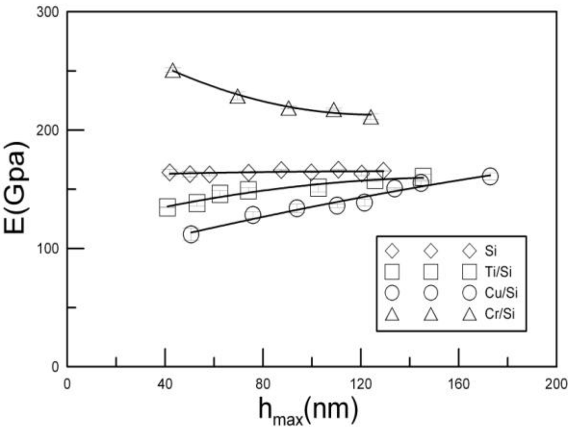

Figure 2: The maximum depth vs. Young's modulus curves of bulk material Si and thin films Ti, Cr and Cu with 300nm thickness deposited on Si substrate by nanoindentation.

3. Numerical simulation

In this investigation, a commercial finite element code ANSYS was used to reproduce the load-depth curves of the Ti, Cu and Cr films on Si substrate (i.e., Ti/Si, Cu/Si and Cr/Si) on the nanoindentation process and the elastic-perfectly plastic material model was considered. An axisymmetric cone with half-included angle of 70.3° which the conical indenter has the same area function as a Berkovich tip was used in this study. The Plan42, Conta171 and Targe169 elements were adopted in finite element analysis. Fig. 3 shows the axisymmetric mesh. As the reported by Lichinchi [3], three-dimensional model was compared with two-dimensional axisymmetric model, no relevant differences are apparent between the two models. Therefore, we have chosen the two-dimensional axisymmetric model in order to restrain the degrees of freedom.

In our work, the Bekervich diamond tip was used and to be simulated as a perfectly elastic material with elastic modulus of 1140 GPa and Poisson' ratio of 0.07. The substrate Si is assumed as a elastic material with Young's modulus of 165 GPa. The thin films Ti, Cu, and Cr were modeled as the elastic-perfectly plastic materials and all the elastic modulus were obtained by nanoindentation and equation (1)-(5) (E_{Ti} =134GPa, E_{Cr} =240GPa, and E_{Cu} =110GPa) and showed in Fig. 2. While the elastic-perfectly plastic material was employed at FEM, the yield stresses ($Y_{Si}, Y_{Ti}, Y_{Cu}, Y_{Cr}$) must be changed for reproducing the experimental load-depth curves and can be obtained as Y_{Ti} =4Gpa, Y_{Cr} =11GPa, and Y_{Cu} =0.75GPa in Fig.4.

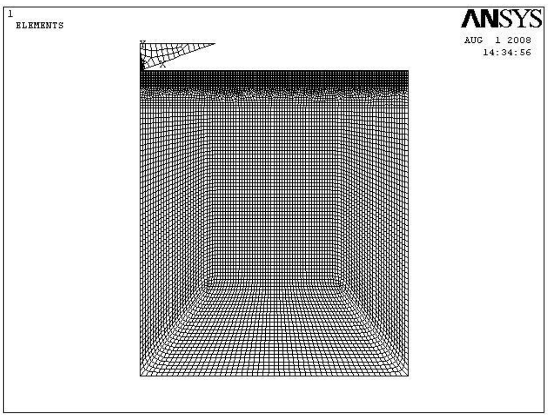

Figure 3: the axisymmetric mesh

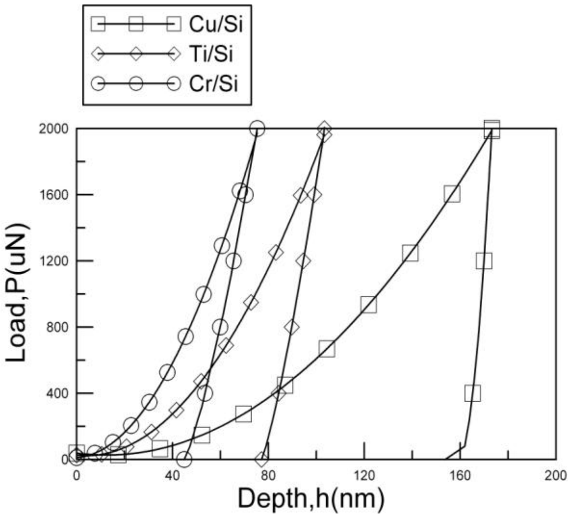

Figure 4: The load-depth curves of 300nm thickness single layer films Ti/Si, Cr/Si, and Cu/Si by FEM.

4. Results and discussion

In this paper, the multilayered coatings on nanoindentation with Berkovich probe tip were investigated with the finite element analysis. The maximum depth vs. the middle layer thickness evaluated from the load-depth curves for the two different types of multilayered coatings Ti/Cu/Si and Ti/Cr/Si by FEM at the thickness of thin film Ti is 300nm, the load is P_{max} =2000uN in Fig. 5. When the middle layer thickness is equal zero, the two multilayered coatings Ti/Cu/Si and Ti/Cr/Si become the case of the single layer Ti/Si. The two curves are at same point while the middle layer thickness is equal zero. For the case of multilayered coatings Ti/Cu/Si, the maximum depth increases with the middle layer thickness Cu increasing. For the case of multilayered coatings Ti/Cr/Si, the maximum depth decreases with the middle layer thickness Cr increasing. Because the Young' modulus and yield stress of the middle layer Cr are larger than the surface layer Ti and the Young' modulus and yield stress of the middle layer Cu are smaller than the surface layer Ti. It is concluded that the maximum depth increases with the softer middle layer thickness increasing but the maximum depth decreases with the harder middle layer thickness increasing.

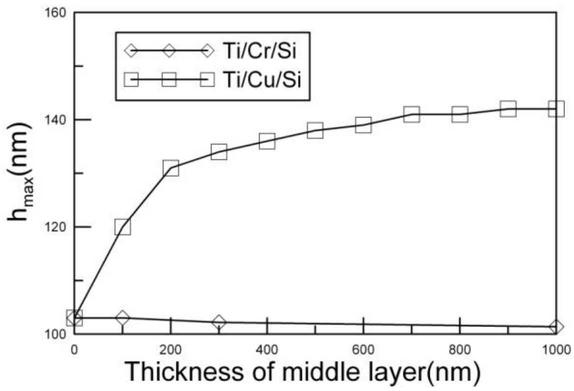

Figure 5: The maximum depth vs. thickness of middle layer of the two different types of multilayered coatings Ti/Cu/Si and Ti/Cr/Si by FEM at the thickness of thin film Ti is 300nm, the maximum load is P_{max} =2000uN.

Fig. 6 presents the load-depth curves of multilayered coatings Ti/Cu/Si by FEM with the surface film Ti 300nm thickness and using difference thickness of middle layer Cu. It shows the loading and unloading curve shifts to the right while increasing the thickness of middle layer Cu. Because the material of middle layer Cu is softer than the surface film Ti. It is concluded that increasing thickness of softer middle layer material is increasing the depth of specimen. We are further to analyze the effect of middle layer, the maximum depth vs. thickness of middle layer evaluated from load-depth data are plotted in Fig. 7. While the maximum depth smaller than 60nm, the maximum depth is not increasing with the thickness of middle layer. Because the penetration depth is not to exceed 20 % of the surface film thickness (the thickness of surface film Ti is 300nm), the effect of middle layer is very small. But when the penetration depth exceed 20 % of the surface film thickness, the maximum depth increases with middle layer thickness increasing. Especially, the larger load P_{max} has larger maximum depth increasing rate.

Finally, the effect of surface layer and middle layer is observed, the maximum depth vs. thickness of different layer is determined from load-depth curves of the multilayered coatings Ti/Cu/Si by FEM, and the load is P_{max} =2000uN in Fig. 8. The curve with the diamond mark represents the thickness of surface layer Ti increases from 300nm to 1000nm as the thickness of middle layer Cu is 300nm. The curve with the square mark represents the thickness of middle layer Cu increases from 100nm to 1000nm as the thickness of surface layer Ti is 300nm. It shows that the maximum depth increases with middle thickness Cu, but the maximum depth decreases with surface thickness Ti. It is very interest, the maximum depth decrease either decreasing middle thickness Cu or increasing surface thickness Ti.

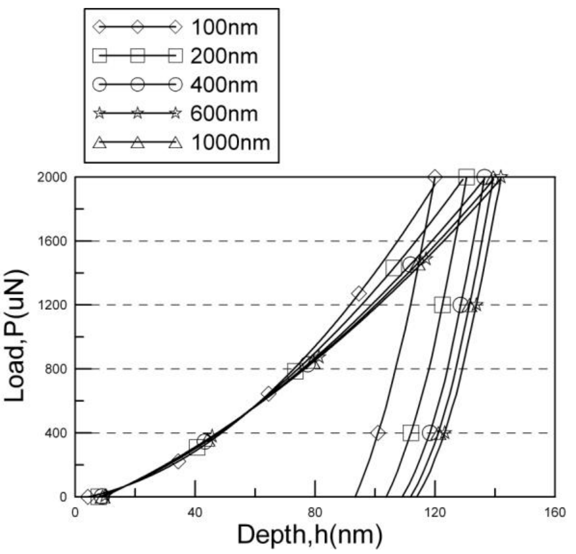

Figure 6: The load-depth curves of multilayered coatings Ti/Cu/Si by FEM in difference thickness middle layer Cu

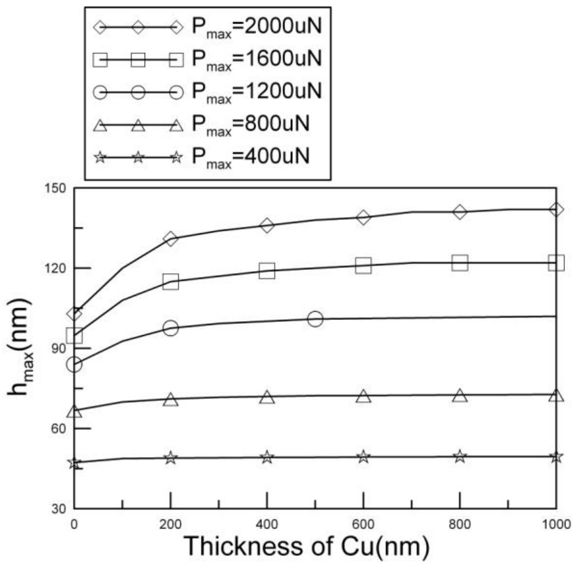

Figure 7: The maximum depth vs. thickness of middle layer of the multilayered coatings Ti/Cu/Si by FEM at the thickness of thin film Ti is 300nm.

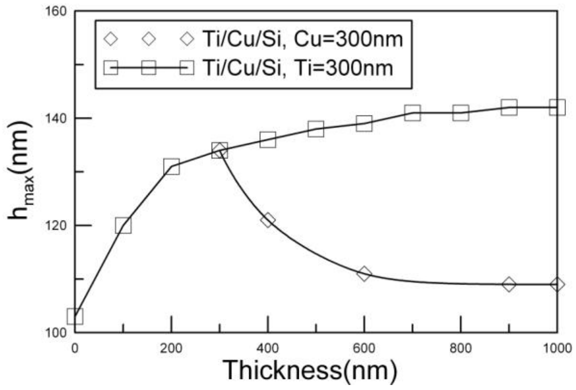

Figure 8: The maximum depth vs. thickness of layer of the multilayered coatings Ti/Cu/Si at the different thickness of surface layer or middle layer, the load is P_{max} =2000uN.

5. Conclusions

Finite element simulations are used to study the mechanical properties of multilayered coatings on nanoindentation with Berkovich probe tip and the following conclusions are obtained.

1. The maximum depth increases with the increasing of softer middle layer thickness but the maximum depth decreases with the increasing of harder middle layer thickness.
2. The lager thickness of soft middle layer material is the softer surface film response.
3. While the penetration depth is not to exceed 20 % of the surface film thickness, the effect of middle layer is very small.
4. The maximum depth decrease either decreasing middle thickness Cu or increasing surface thickness Ti at the multilayered coatings Ti/Cu/Si.

6. References

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