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NANOINDENTATION OF THIN-FILM-SUBSTRATE SYSTEM: DETERMINATION OF FILM HARDNESS AND YOUNG'S MODULUS*

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ABSTRACT: In the present paper, the hardness and Young's modulus of film-substrate systems are determined by means of nanoindentation experiments and modified models. Aluminum film and two kinds of substrates, i.e. glass and silicon, are studied. Nanoindentation XP II and continuous stiffness mode are used during the experiments. In order to avoid the influence of the Oliver and Pharr method used in the experiments, the experiment data are analyzed with the constant Young's modulus assumption and the equal hardness assumption. The volume fraction model (CZ model) proposed by Fabes et al. (1992) is used and modified to analyze the measured hardness. The method proposed by Doerner and Nix (DN formula) (1986) is modified to analyze the measured Young's modulus. Two kinds of modified empirical formula are used to predict the present experiment results and those in the literature, which include the results of two kinds of systems, i.e., a soft film on a hard substrate and a hard film on a soft substrate. In the modified CZ model, the indentation influence angle, φ, is considered as a relevant physical parameter, which embodies the effects of the indenter tip radius, pile-up or sink-in phenomena and deformation of film and substrate.

KEY WORDS: nanoindentation, hardness, Young's modulus, film-substrate system

1 INTRODUCTION

Several methods have been developed to determine the mechanical properties of engineering materials, such as Young's modulus, yield stress and hardness. In recent years, the indentation methods have been much favored due to their many advantages over the conventional methods. Most importantly, these methods, particularly, the nanoindentation test, can distinguish the deformations of individual components in film-substrate systems. It offers a potential means of calculating the individual properties of the thin film and substrate. However, in practice, the explanation of this process is far from straight forward and is currently attracting much research interest. In order to obtain intrinsic film properties from large indentations, one needs to understand how mechanical properties of the substrate affect measurements of film stiffness and hardness.

Several theoretical methods have been developed to describe the hardness of film-substrate systems and some models are proposed[1~9]. These models are based on the idea that the composite hardness is determined by the weighted average of film and substrate hardnesses in proportion to the relative deformed areas or volumes. One typical model proposed by Jonsson and Hogmark[5], in which it is assumed that the composite hardness is determined by the intrinsic hardness of the film and the intrinsic hardness of the substrate in proportion to the projected area of the film and the area of the substrate contributing to the total projected area, which is left after unloading. Another typical model is the volume model proposed by Fabes et al.[7], in which it is assumed that the deformed regions are of cone shape. The radius of the cone basis at the surface is taken to be equal to the

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effective radius of the plastic imprint and half of the cone angle at the tip is 45°.

In order to analyze and obtain the elastic modulus of thin films, several theoretical methods[9~11] have been established and many experimental methods have been developed, such as bending of microbeams, nanoindentation, as is reviewed in Refs.[12~14]. The nanoindentation is advantageous because it has no special requirements for the specimen shape and preparation. However, during the indentation, the deformation of the film-substrate system is very complex, not only because the film and substrate have different mechanical properties but also because the deformation ratio of the film to the substrate and the influence of the substrate will change according to the indentation depth. The measured value is a composite modulus including the influences of the instrument, film and substrate. The film modulus must be determined by a suitable treatment of the data. For the Oliver and Pharr method[15] used in the nanoindentation test, the low precision on the contact area will influence the reduced modulus. The assumption that the hardness calculated using the constant Young's modulus assumption is also the hardness of the same film on the other substrate is used to obtain the reduced elastic modulus.

In the present study, nanoindentation experiments on Al/Si and Al/glass are carried out and the mechanical properties of film-substrate systems, especially the hardness of the films and the Young's modulus are investigated. Since aluminum and glass have similar Young's modulus, the constant Young's modulus assumption is adopted and the composite hardness will be obtained. Since aluminum films on silicon and glass substrates have the same thicknesses, we assume that the aluminum films have the same hardness on different substrates, from which we can also obtain the Young's modulus of Al/silicon systems. Two modified models are proposed and used to analyze the measured hardness and Young's modulus, respectively.

2 NANOINDENTATION EXPERIMENTS AND RESULTS

Al films are prepared on glass and silicon substrates by sputtering and the base pressure in the chamber prior to sputtering is 5×10^{-7} Torr. The sputtering pressure is 1.0 Pa. Three kinds of nominal thicknesses of Al films are 52.3 nm, 244.7 nm and 850.9 nm, respectively. The deposition rates are 110 Å/min, 160 Å/min and 160 Å/min, respectively and all the sputtering processes are under 1 000 w of power. Since the thinnest Al film is only 52.3 nm thick, the experiment results have a fluctuation due to the influence of many undetermined factors and the results are not given in the present paper.

The mechanical properties of the substrates and films are to be determined by using Nanoindentation XP II with a Berkovich indenter tip. The continuous stiffness mode is used in all experiments. The indentations are made with a constant nominal strain rate $0.05\,\mathrm{s}^{-1}$. Five indentation points are chosen in each sample and the results presented are an average of these five indentations. Hardness and Young's modulus are measured directly by means of the Oliver and Pharr analysis method[15]. It should be noted that the Oliver and Pharr's method is only for monolithic material and only for sink-in. In order to avoid the calculation of the contact area, which sometimes causes some deviations in the results due to sink-in or pile-up, we will also use the method given by Joslin and Oliver[16] to analyze the experiment data.

With depth-sensing nanoindentation devices, elastic modulus is determined from

$$E_\mathrm{r} = \frac{\sqrt{\pi}}{2\beta} S \frac{1}{\sqrt{A}} = \frac{\sqrt{\pi}}{2\beta} \frac{\mathrm{d}P}{\mathrm{d}h} \frac{1}{\sqrt{A}} \tag{1}$$

$$S = \frac{2\beta}{\sqrt{\pi}} \sqrt{A} E_\mathrm{r} \tag{2}$$

where A is the projected area of the contact, β is a constant that depends on the geometry of the indenter. $S = \mathrm{d}P/\mathrm{d}h$ is the slope of the load-displacement curve at the beginning of the unloading and E_r is the reduced Young's modulus.

If the film has a similar Young's modulus with the substrate, i.e., $E_\mathrm{f} \approx E_\mathrm{s}$, then the reduced Young's modulus can be written as

$$\frac{1}{E_\mathrm{r}} = \frac{1-\nu_\mathrm{i}^2}{E_\mathrm{i}} + \frac{1-\nu_\mathrm{f}^2}{E_\mathrm{f}} \tag{3}$$

where E_i, E_f are Young's moduli of the indenter and film, respectively. ν_i, ν_f are Poisson's ratios of the indenter and film, respectively.

Hardness is usually defined as

$$H = \frac{P}{A} \tag{4}$$

Eliminating contact area from Eqs.(1) and (4), the composite hardness for the film-substrate system is obtained as

$$H = \frac{4\beta^2}{\pi} \frac{P}{S^2} E_\mathrm{r}^2 \tag{5}$$

From the above equation, one can see that the composite hardness, H, is directly proportional to the parameter, P/S^2, and proportional to the square of the reduced modulus, E_r^2. If the film has a similar Young's modulus with that of the substrate, the reduced modulus, E_r will approximately be a constant, which is called the constant Young's modulus assumption.

As pointed out by Ref.[17] that there is a problem existing in determining the intrinsic hardness of thin films, that is, the measurement is influenced by the properties of the substrates, especially, when the film is very thin. The hardness measurement of a soft film is enhanced by the hard substrate while the hardness of a hard film is reduced by the soft substrate. True contact area cannot be obtained precisely during the experiment because all the measurements are based on the Oliver and Pharr method. The true contact depth is underestimated for a soft film on a hard substrate system and overestimated for a hard film on a soft substrate system. Thus, the hardness is overestimated for a soft film on a hard substrate and underestimated for a hard film on a soft substrate.

In order to avoid the calculation of the true contact area, the method proposed by Joslin and Oliver[17], Eq.(5), is used in the present paper. The parameter, P/S^2, can be used only when the material is elastically homogeneous and the Young's modulus of the indenter is known.

The Young's modulus of glass is measured by nanoindentation. The reduced Young's modulus of Al/glass system measured almost keeps a constant, so we take the Young's modulus of Al as $E_{\text{Al}} = E_{\text{glass}}$. In the test, it is assumed that Poisson's ratio $\nu_{\text{Al}} = \nu_{\text{glass}} = 0.3$. The Young's modulus of the Berkovich indenter is taken as 1 140 GPa and the Poisson's ratio is 0.07, which is taken from literature. The constant β is taken as 1.034, corresponding to the Berkovich indenter geometry. According to Eq.(5), the hardness is determined from the load, P, and the contact stiffness, S, through the parameter, P/S^2. Figure 1 shows the composite hardness as a function of the normalized indentation depth for Al/glass systems with two different film thicknesses.

From Fig.1, one can see that at a very small indentation depth, the composite hardness decreases as the indentation depth increases, which is due to the size effects. At a deeper indentation depth the hardness gets to a constant value of about 1.1 GPa for the 244.7 nm film and 0.9 GPa for the 850.9 nm film. The hardness is approximately constant until the indentation depth is about 0.6 times the film thickness. Then the hardness starts to increase with increasing indentation depth. When the indentation gets to the film-substrate interface, a more significant increase in hardness with the increase of indentation depth can be found, which is caused mainly by the indenter penetration into the harder substrate. From the analysis, we take the plateau values to be the mechanical properties of the film.

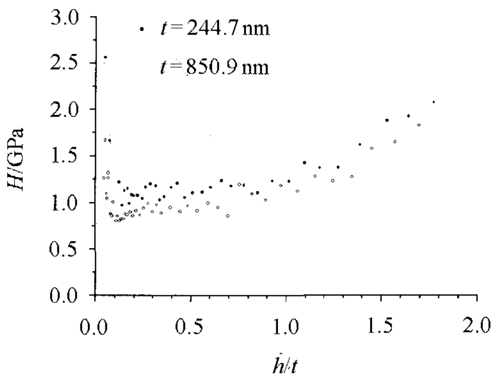


Fig.1 Plot of the composite hardness versus the normalized indentation depth using the constant Young's modulus assumption for Al/glass systems with two different thicknesses

Figure 2 is a plot of the parameter, P/S^2, which is used by Joslin and Oliver[16] to avoid the calculation of the contact area and to obtain a more precise hardness value, versus the normalized indentation depth, h/t, for Al/silicon systems with film thicknesses 244.7 nm and 850.9 nm. After initial drop, which can be attributed to an indentation size effect, the value of P/S^2 increases inversely with the thickness of the film, that is, the value of P/S^2 is a little larger for Al/silicon with thickness 244.7 nm than that with thickness 850.9 nm, which is due to the different hardness for films with different thicknesses.

It is reasonable according to Eq.(5), that is, P/S^2 is proportional to the hardness of the composite

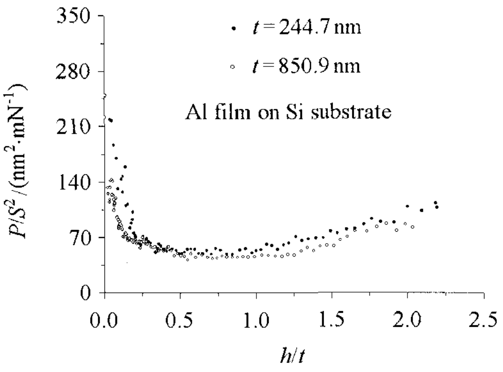


Fig.2 Plot of P/S^2 versus the normalized indentation depth, h/t

hardness

$$\frac{P}{S^2} = \frac{\pi}{4\beta^2}\frac{H}{E_r^2} \tag{6}$$

In the present experiments, Al films are deposited on substrates, which has a similar modulus as the film (glass) or is stiffer (silicon) than Al film. Hence we assume that the film accommodates all the plastic deformation and the substrate begins to yield only when the indenter is close to the film-substrate interface. When the films on different substrates, i.e., Al/glass and Al/ Si, have equal thicknesses, we assume that the hardness calculated for the Al/glass films using the constant Young's modulus assumption is also the hardness of Al films on silicon substrate, which is called the equal hardness assumption. Then, the reduced modulus for the Al films on silicon substrates can be calculated from P/S^2 using the following equation

$$E_r = \sqrt{\frac{\pi}{4\beta^2}\frac{S^2}{P}H(E)} \tag{7}$$

where $H(E)$ is the hardness , which is obtained from the constant Young's modulus assumption.

Figure 3 shows the reduced Young's modulus of Al/silicon system, E_r, versus the normalized indentation depth, h/t. From Fig.3 one can see that Young's moduli for Al films with different thicknesses are almost equal, which means that Young's moduli for the same material films are independent of the film thicknesses. The reduced modulus increases with increasing indentation depth due to the substrate effect.

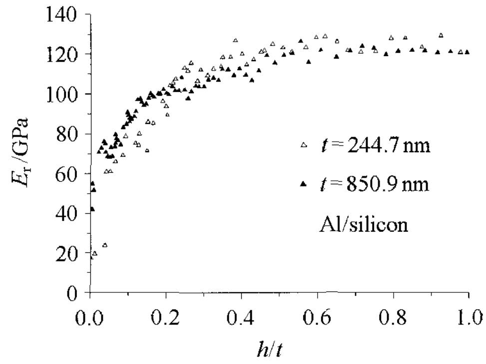


Fig.3 Reduced Young's modulus, E_r, of Al/silicon as a function of the normalized indentation depth, h/t

3 ANALYSIS OF FILM HARDNESS

The volume fraction model is a model for interpreting nanoindentation hardness of thin films proposed by Fabes et al.[7] and the deformed volume is assumed to be of cone zone shape. Three stages are identified during the indentation process: (1) in stage I, both the indenter and its associated plastic strain field are assumed to be confined to the film. In this stage, the hardness of the film is given simply by the measured hardness and is independent of the indentation depth; (2) In stage II, the indenter is still in the films, but the associated plastic strain field has penetrated into the substrate and the composite hardness is influenced by the deformed volume in the substrate, V_s; (3) In stage III, the indenter has penetrated into the substrate and the hardness is influenced by the sum of the material volume deformed directly by the indenter, V_{sd}, and that deformed by the transfer across the film-substrate interface, V_s.

Since the deformed volume appears in both the numerator and denominator in Fabes et al.'s formula, the exact geometric shape (sphere, cone etc.) of the volume should not be important. They maintained that only the ratio of the volumes were important and assumed that $\varphi = 45°$ for the half triangular cone, as shown in Fig.4.

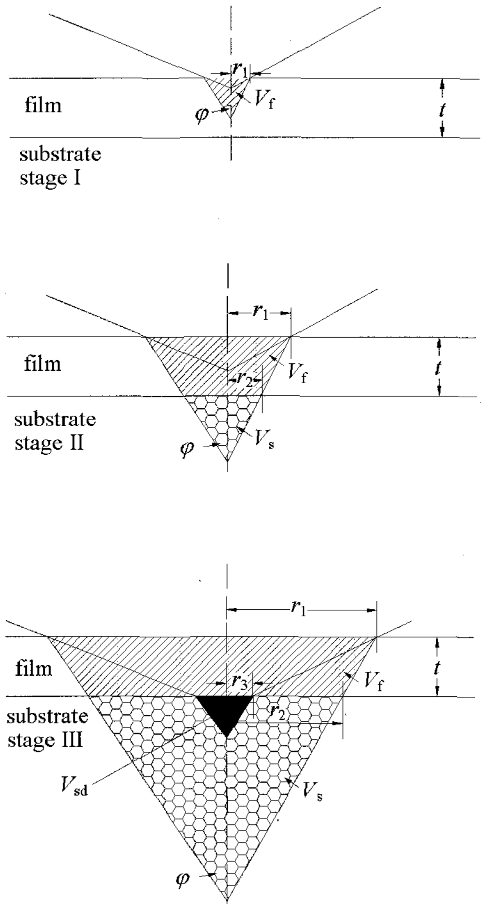


Fig.4 Schematic of dimensions for modified CZ model

But Ref.[8] pointed out that the CZ model failed to predict the composite hardness for a hard film on a soft substrate by underestimating the contribution of the substrate. The reason for this is owing to the fixed conical shape of the plastic zones, which does not depend on the relation between the elastic-plastic properties of the film and the substrate. Thus the transitional value, h/t, between stage I and stage II keeps unchanged though the relation between properties of the film and the substrate changes, which means that the transitional values, h/t, between stage I and stage II are the same for a soft film on a hard substrate system and that of a hard film on a hard substrate. However, many experiments have shown that the substrate effect influences the composite hardness sooner for a hard film on a soft substrate system as compared to that of a soft film on a hard substrate. In the CZ model, the cone shape angle is fixed to be 45° for both kinds of systems, so the results of both a soft film on a hard substrate and a hard film on a soft substrate can not be correctly predicted simultaneously.

From the above analysis, it can be seen that the CZ model should be modified in order to describe both a soft film on a hard substrate and a hard film on a soft substrate. An improvement may be to change the cone tip angle, which will change the transition value between stage I and stage II. A modified CZ model is proposed in the present paper. In the modified CZ model, the cone shape of the deformation zone keeps unchanged but the cone tip angle φ becomes an undetermined parameter. φ is determined according to the relation between the elastic-plastic properties of the film and the substrate.

Three stages are also identified according to indentation depth and the film thickness as shown in Fig.4. Since the continuous stiff mode is used in the present experiment, we will use directly the indentation depth, h, and the area is expressed as

$$A = 24.5h^2 \tag{8}$$

In stage I, the corresponding radius, r_1, is related to the total indentation depth as

$$r_1 = \sqrt{\frac{A}{\pi}} \tag{9}$$

$$H^{\mathrm{I}} = H_{\mathrm{f}} \tag{10}$$

In stage II, the separate contributions to the composite hardness from the film and the substrate are determined. The deformed volume is

$$V_{\mathrm{f}} = \frac{1}{3}\pi t(r_1^2 + r_2^2 + r_1 r_2) \tag{11}$$

$$r_2 = r_1 - t\tan\varphi \tag{12}$$

$$V_{\mathrm{s}} = \frac{1}{3}\pi r_2^3/\tan\varphi \tag{13}$$

Then the hardness can be expressed as

$$H^{\mathrm{II}} = \frac{H_{\mathrm{f}}V_{\mathrm{f}}\xi^{\alpha} + H_{\mathrm{s}}V_{\mathrm{s}}}{V_{\mathrm{f}}\xi^{\alpha} + V_{\mathrm{s}}} \tag{14}$$

where

$$\xi = \frac{E_{\mathrm{f}}H_{\mathrm{s}}}{E_{\mathrm{s}}H_{\mathrm{f}}} \tag{15}$$

with α as an adjustable parameter.

In stage III, the indenter itself is penetrated into the substrate. In this stage the deformation volume of the film is still given by Eq.(11), and the deformation volume transmitted from the film to the substrate is still given by Eq.(13). However, there is an additional contribution to the substrate volume from material that is deformed directly by the indenter. This volume is defined as V_{sd}

$$V_{\mathrm{sd}} = \frac{1}{3}\pi r_3^3/\tan\varphi \qquad r_3 = (h-t)\tan\varphi_1 \tag{16}$$

$$H^{\mathrm{III}} = \frac{H_{\mathrm{f}}V_{\mathrm{f}}\xi^{\alpha} + H_{\mathrm{s}}V_{\mathrm{s}} + H_{\mathrm{s}}V_{\mathrm{sd}}}{V_{\mathrm{f}}\xi^{\alpha} + V_{\mathrm{s}} + V_{\mathrm{sd}}} \tag{17}$$

where V_{f}, V_{s}, V_{sd}, r_1, r_2 and r_3 are shown in Fig.4, φ_1 is a half of the indenter tip angle and in this paper it takes the value $\varphi_1 = 70.3°$, corresponding to the Berkovich indenter.

Qualitative analysis of the influence of φ is shown in Fig.5, from which one can see that when the value φ increases, the transitional value, h/t, between stage I and stage II, will increase, which means that the substrate effect influences the composite hardness much later for a larger φ.

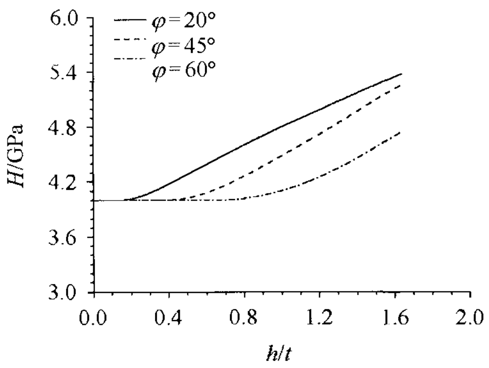


Fig.5 Hardness versus the normalized depth for a qualitative analysis of the cone shape angle, φ ($H_{\mathrm{f}} = 4.0$ GPa, $H_{\mathrm{s}} = 8.2$ GPa, $E_{\mathrm{f}} = 88.5$ GPa, $E_{\mathrm{s}} = 88.5$ GPa, $\alpha = 0.9$)

The value of φ for a soft film on a hard substrate should be larger than that for a hard film on a soft substrate since the soft substrate is easier to yield and the substrate effect comes sooner.

Using the modified CZ model, we can predict the composite hardness obtained in the present experiment. The predicted composite hardness values are plotted in Fig.6, in which the hardness of the glass substrate is 8.2 GPa, the parameter, α takes the value of 0.9. For the two kinds of film thicknesses, 244.7 nm and 850.9 nm, the film hardness is 1.1 GPa and 0.9 GPa, respectively and the deformation volume conical angle is $\varphi = 45°$.

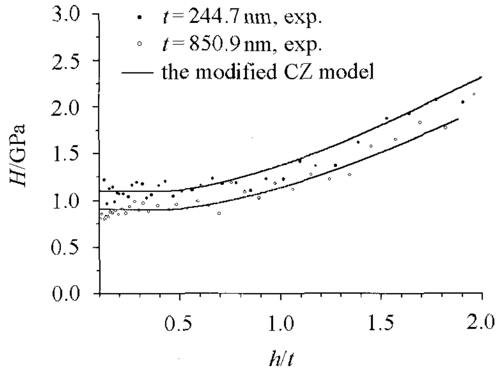
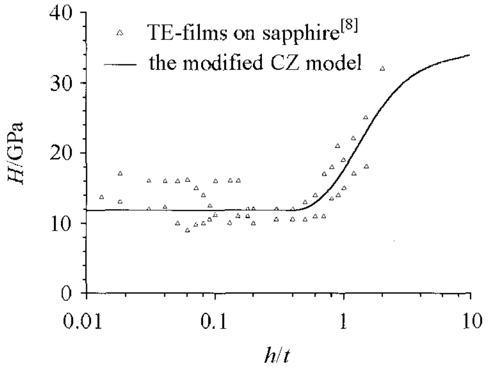


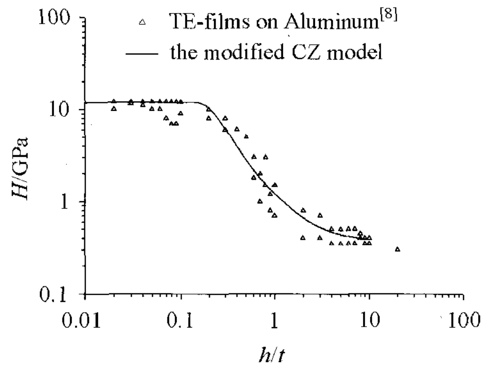
Fig.6 Hardness as a function of the normalized depth for Al/glass systems with the experiment results of the present paper and theoretical results of the modified CZ model

From Fig.6, one can see that the transition region is about $h/t = 0.6$, which is consistent with the experiment results. The film-substrate system, Al/glass, is a soft film on a hard substrate and the modified CZ model can describe the experiment data well.

In order to verify further the modified CZ model, we consider the experiment data in Ref.[8], which are Al_2O_3 films on aluminum and sapphire substrates. In Ref.[8], there are two kinds of methods to produce the Al_2O_3 film, one is anodic oxidation of polycrystalline Al (AO films) and the other is thermal evaporation of Al in an oxygen atmosphere onto the different substrates (TE films). Here we consider TE films on aluminum and sapphire substrates to verify the modified CZ model since one system corresponds to a soft film on a hard substrate and the other is a hard film on a soft substrate. We only use the minimum and maximum experiment data at each normalized indentation depth since there are many experiment data for different film thicknesses at each normalized depth. In the experiment data, one can see that the transition values are much different for the two film-substrate systems and for one system $h/t \approx 0.2 \sim 0.3$; for the other $h/t \approx 0.5 \sim 0.6$. The predicted results and the experiment data in Ref.[8] are plotted in Fig.7, from which one can see that the theoretical results are consistent well with the experiment results for both kinds of systems. The corresponding values of parameter, φ, are 45° and 20°, respectively. One can see that the parameter, φ, takes the value of 45° for a soft film on a hard substrate, which also proves that the CZ model can describe this kind of film-substrate system well. The parameter, φ, for a hard film on a soft substrate takes the value of 20°, which needs more experiments to verify.



(a)



(b)

Fig.7 The composite hardness as a function of the normalized depth: (a) TE film on sapphire with experiment results taken from Ref.[8] and the results of the modified CZ model; (b) TE film on aluminum with experiment results taken from Ref.[8] and the results of the modified CZ model

4 ANALYSIS OF THE REDUCED YOUNG'S MODULUS

Several theoretical methods have been developed for explaining the reduced modulus and determining the elastic modulus of thin films. In order to analyze the theoretical formula and explain the experiment results, several theoretical methods have been used in the present paper.

The relative penetration is often characterized by the ratios h/t, h_c/t or a/t, where a is the contact radius and t is the film thickness. Due to the continuous stiffness mode used in the present paper, the relative penetration, h/t, is chosen, where h is the indentation depth. Theoretical formula are given as follows.

4.1 Existing Formula

4.1.1 Linear Formula

The linear function is as follows

$$\frac{1}{E_{\mathrm{r}}}=\frac{1}{E}+\frac{1-\nu_{\mathrm{i}}^2}{E_{\mathrm{i}}} \tag{18}$$

$$E=\frac{E_{\mathrm{f}}}{1-\nu_{\mathrm{f}}^2}+\Big(\frac{E_{\mathrm{s}}}{1-\nu_{\mathrm{s}}^2}-\frac{E_{\mathrm{f}}}{1-\nu_{\mathrm{f}}^2}\Big)\frac{h}{t} \tag{19}$$

where E_{r} is the reduced modulus, E_{s}, E_{f} and E_{i} are Young's modulus of the substrate, film and the indenter, respectively.

4.1.2 Gao's Formula

In Gao's original formula, the relative penetration is described by a/t, where a is the contact radius. In the present paper, we use h/t. E is obtained as follows

$$E=\frac{E_{\mathrm{s}}}{1-\nu_{\mathrm{s}}^2}+\Big(\frac{E_{\mathrm{f}}}{1-\nu_{\mathrm{f}}^2}-\frac{E_{\mathrm{s}}}{1-\nu_{\mathrm{s}}^2}\Big)\cdot\Big\{\frac{2}{\pi}\arctan\frac{t}{h}+\frac{1}{2\pi(1-\nu)}\Big[(1-2\nu)\frac{t}{h}\ln\Big(1+\frac{h^2}{t^2}\Big)-\frac{ht}{h^2+t^2}\Big]\Big\} \tag{20}$$

where ν is the effective Poisson's ratio. The difference in the Poisson's ratio of the film and substrate has a small effect on the reduced modulus, which is used in Ref.[17]. In the present paper, the Poisson's ratios of the film and the substrate are assumed to be equal, so the effective Poisson's ratio $\nu=\nu_{\mathrm{s}}=\nu_{\mathrm{f}}$.

4.1.3 Exponential Formula

In this formula, E is described as

$$E=\frac{E_{\mathrm{s}}}{1-\nu_{\mathrm{s}}^2}+\Big(\frac{E_{\mathrm{f}}}{1-\nu_{\mathrm{f}}^2}-\frac{E_{\mathrm{s}}}{1-\nu_{\mathrm{s}}^2}\Big)\mathrm{e}^{-\alpha h/t} \tag{21}$$

where α is a constant to be adjusted.

4.1.4 Doerner and Nix Formula

An empirical relationship (DN formula) among E_{i}, E_{f} and E_{s} was proposed by Doerner and Nix[10], with a structure slightly different from the above theoretical formula. The moduli are replaced by the corresponding reciprocals, i.e., compliances, as follows

$$\frac{1}{E_{\mathrm{r}}}=\frac{1-\nu_{\mathrm{i}}^2}{E_{\mathrm{i}}}+\frac{1-\nu_{\mathrm{f}}^2}{E_{\mathrm{f}}}\big(1-\mathrm{e}^{-\alpha t/h}\big)+\frac{1-\nu_{\mathrm{s}}^2}{E_{\mathrm{s}}}\mathrm{e}^{-\alpha t/h} \tag{22}$$

where α is an adjustable parameter.

4.2 Comparison with Experiment Results

In the present paper, the relative penetration variable, h/t_{eff}, is also used for analysis in all the above formula, where the effective thickness, t_{eff}, can be obtained from the identity of stressed film volumes

$$\pi a^2 t_{\mathrm{eff}}=\pi a^2 t-\frac{1}{3}\pi a^2 h \tag{23}$$

Then

$$t_{\mathrm{eff}}=t-\frac{1}{3}h \tag{24}$$

Another method, which is a phenomenological extension of the formula of Saha and Nix[17], is proposed by Jager [18], but only one case of the experiment in Ref.[17] is analyzed and compared with the formula.

Here, in order to test the theoretical methods for determining elastic modulus of thin films, the experiment results of Al/silicon and Al/sapphire with two kinds of film thicknesses, 0.5 μm and 2 μm given by Ref.[17] are used. The above four theoretical formula are compared with the experiment results. Both h/t and h/t_{eff} are used to compare the theoretical and experiment results.

Young's modulus of Al film is taken as 73 GPa and the moduli of silicon and sapphire are taken as 172 GPa and 440 GPa, respectively, which are the same as those in Ref.[17]. Here, only α is an adjustable parameter and the film modulus is not a parameter to be adjusted.

From the comparison between the four formula and the experiment results, we find that only the exponential formula can describe the experiment data well for both 0.5 μm and 2 μm film systems. The linear formula cannot give satisfactory results, especially when the indentation depth is large. The reason is that the thickness of the film beneath the indenter is smaller than its original value, since (a) ductile films on hard substrates become thinner due to plastic flow during loading, and (b) when E is determined, the contact surface is still under a high pressure and the film is also significantly compressed elastically. Thus, the use of the original film thickness t can cause a systematic shift. Gao's formula can give the correct trends between the indentation depth and the reduced modulus but the deviation is large at a large indentation depth, because Gao's formula for determining the film modulus is an analytical solution for the contact of a rigid cylindrical punch with a semi-infinite elastic body with a film. Doerner and Nix's formula (DN formula) give good results for $2\,\mu$m film system but it cannot describe the $0.5\,\mu$m film system well, especially at a small indentation depth.

The film modulus is assumed to take values from the referenced work[17] and then compare the theoretical results with the experiment data, which is also an effective method to study the validity of the theoretical formula. Once we find a valid theoretical formula, the film modulus can be obtained to compare with the experiment data.

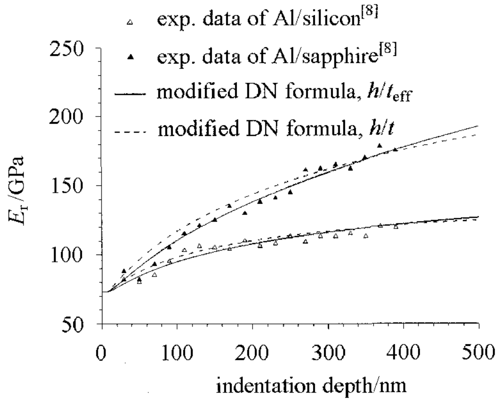
4.3 Modified DN Formula

According to the experiment results in Ref.[17], we propose an empirical formula, which is a modified DN formula as given below.

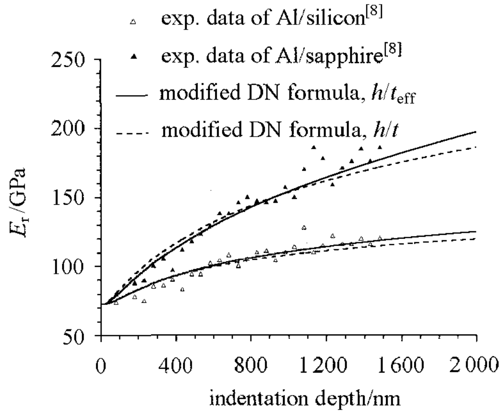
$$\frac{1}{E_{\mathrm{r}}} = \frac{1-\nu_{\mathrm{i}}^2}{E_{\mathrm{i}}} + \frac{1-\nu_{\mathrm{f}}^2}{E_{\mathrm{f}}}\left[1-\mathrm{e}^{-\alpha(t/h)^{2/3}}\right] + \frac{1-\nu_{\mathrm{s}}^2}{E_{\mathrm{s}}}\mathrm{e}^{-\alpha(t/h)^{2/3}} \tag{25}$$

Comparing Doerner and Nix formula and Eq.(25), one can see that only the exponent, 2/3, is different, which is an optimized parameter. It means that Doerner and Nix's model underestimates the substrate effects at a small indentation depth and overestimates the substrate effects at a large indentation depth, while comparing with Eq.(25), as commented in Ref.[18].

Using the empirical formula, Eq.(25), we analyze the experiment data given by Saha and Nix[17] as shown in Fig.8.



(a)



(b)

Fig.8 Comparisons of the experiment data for (a) $0.5\,\mu$m and (b) $2\,\mu$m Al films on silicon and sapphire substrates given in Ref.[17] with the modified DN formula

The moduli of Al film and silicon and sapphire substrates are taken as 73 GPa, 172 GPa and 440 GPa in Ref.[17], only the parameter, α, is adjustable. Both the results for h/t and h/t_{eff} are given. From Fig.8, one can see that the empirical formula can give satisfactory results for both thin and thick film systems. The result corresponding to the parameter h/t_{eff} is better than that for h/t, comparing with the experiment data.

Using this empirical formula, we also analyze the experiment data in the present paper as shown in Fig.9, from which one can see that both the theoretical results and the experiment results agree well with each other. In Fig.9, the experiment data are obtained through the constant Young's modulus assumption. Here h, not h_{c}, is used to calculate the

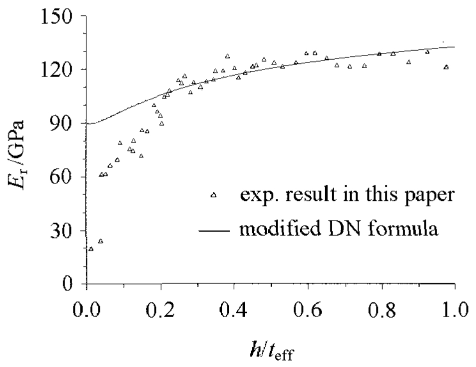


Fig.9 Comparison of the experiment data for 244.7 nm and 850.9 nm Al films on silicon substrates given in the present paper with the modified DN formula

area, the adjustable parameter, α, will contain the influence of the depth and the influences of sink-in or pile-up. More experiment results are needed to verify the empirical formula.

5 CONCLUSIONS

In the present paper, nanoindentation experiments on the hardness and Young's modulus of Al/glass and Al/silicon have been carried out. The hardness is strongly influenced by the hardness of the substrate beyond a transition value, i.e., the hardness increases with increasing indentation depth for a soft film on a hard substrate beyond an indentation depth. For the systems with the same material films but different film thicknesses, the film hardness is different, which has been explained in Refs.[19~20].

A modified CZ model is proposed, in which a conical deformation volume shape is assumed, but the conical tip angle is undetermined. The conical tip angle contains the influences of pile-up or sink-in since the total depth is used in the modified CZ model. Also, the conical tip angle embodies the degree of the substrate effects on the composite hardness and determines the transitional value between stage I and stage II. Due to the introduction of the conical angle, the modified CZ model could describe both a soft film on a hard substrate system and a hard film on a soft substrate system.

From many experiment results, one can see that the substrate effect shows itself sooner in a hard film on a soft substrate system as compared to a soft film on a hard substrate, which means that the plastic deformation appears in the substrate earlier in the former as compared to the latter, which is proved by the value of the conical angle, at least in TE films on aluminum and TE films on sapphire systems[8].

Young's modulus has been measured and it is found that the Young's modulus has no relation with the film thickness, but the reduced Young's modulus is influenced greatly by the substrate effect.

Four kinds of theoretical formula determining the film modulus are investigated. The model proposed by Doerner and Nix[10] has been modified in order to give satisfactory results comparing the experiment data in Ref.[17].

Using the modified model, the experiment data in the present paper are studied and both the theoretical results and the experiment results are consistent well with each other, which demonstrates that the modified DN model is effective. On the other hand, from the comparison results, one can see that the result using the penetration ratio h/t_{eff} is better than that using h/t.

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