

Analysis of continuous stiffness data measured during nanoindentation of titanium films on glass substrate

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ABSTRACT

An empirical function was proposed to describe the continuous stiffness curves, i.e., E_r^2/H versus h (where E_r is the reduced modulus, H is the composite hardness and h is the indenter penetration depth), measured with nanoindentation tests for titanium films on glass substrate. By analyzing the variations of the parameters included in this empirical equation with film thickness, the physical meanings of this empirical equation were discussed. It was shown that the mechanical properties of the substrate and the film may be extracted from such analyses.

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1. Introduction

The continuous stiffness measurement (CSM) is a widely adopted nanoindentation test, in which a small harmonic force is added on the indenter, and the harmonic response of the indenter is measured at the excitation frequency [1–3]. The main advantages of CSM is that it offers the direct measurement of dynamic contact stiffness, S , at any point along the loading curve. Furthermore, CSM is insensitive to thermal drift and mechanical results are not considerably influenced by drift errors [4].

With the CSM, the hardness and the Young's modulus of a film–substrate system can be determined continuously as functions of indenter penetration depth. Many authors have tried to discuss the effect of substrate on the mechanical properties of the film–substrate system by directly analyzing the observed penetration-depth-dependence of the hardness and the Young's modulus, and then extracting the mechanical properties of the films [5–8].

Another approach to analyze the measured continuous stiffness curve is to express the experimental data measured from the CSM by plotting P/S^2 , where P is the indentation load and S the contact stiffness, as a function of the indenter penetration depth [9–11]. The theoretical basis for this approach was established by Joslin and

Oliver [12], and then developed by Saha and Nix [13]. The principle of this approach can be generalized as follows.

Hardness is usually defined as

$$H = \frac{P}{A} \quad (1)$$

where A is the contact area.

For the nanoindentation test, Young's modulus is determined from [1]

$$S = \frac{dP}{dh} = \frac{2\beta}{\sqrt{\pi}} E_r \sqrt{A} \quad (2)$$

where β is a constant dependent on the geometry of the indenter ($\beta = 1.034$ for a Berkovich indenter), and E_r is the reduced modulus given by

$$\frac{1}{E_r} = \frac{1 - \nu_i^2}{E_i} + \frac{1 - \nu_f^2}{E_f} \left[1 - \exp\left(-\alpha \frac{t}{\sqrt{A}}\right) \right] + \frac{1 - \nu_s^2}{E_s} \exp\left(-\alpha \frac{t}{\sqrt{A}}\right) \quad (3)$$

where the subscripts i , f and s represent the indenter, the film and the substrate, respectively, ν is the Poisson's ratio, α is a constant related to the indenter geometry, and t is the film thickness under the indenter.

Eliminating the contact area, A , from Eqs. (1) and (2), one can obtain the composite hardness for a film–substrate system [12],

$$\frac{P}{S^2} = \frac{\pi}{4\beta^2} \frac{H}{E_r^2} \quad (4)$$

Thus, we can find that analysis of the variation of P/S^2 with indentation penetration depth may provide some useful information of the mechanical properties of the test specimen.

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In general, the composite hardness, H , and the reduced modulus, E_r , can be calculated with the experimental data obtained from CSM [5–8]. Therefore, it seems to be more convenient to study the mechanical properties of the test material by directly establishing and analyzing the E_r^2/H as a function of the penetration depth.

In this paper, we will propose an empirical function to describe the experimentally observed variation of E_r^2/H with indenter penetration depth, h . Titanium films on glass substrate with different thicknesses were selected for this study. It will be shown that, with this empirical equation, some useful information of the mechanical properties of the film and the substrate may be obtained.

2. Experimental

The materials examined in this study are a series of titanium films on commercial soda-lime glass substrate prepared by a mid-frequency magnetron sputtering system (SP0806AS, Beijing Powertech Co. Ltd.) with dual pure titanium targets. Before the deposition of the films, the chamber was pumped out up to 2.0×10^{-3} Pa. Ultra-sonically clean soda-lime glass substrates were sputtered by Ar^+ for 10 min. During the deposition of the films, the working pressure was fixed at about 0.5 Pa, the target current was 10 A, and the bias voltage of the substrate was -100 V. The only parameter changed during deposition was the deposition time, which was in the range from 15 to 240 min, so as to obtain films with different thicknesses but of the same quality. It was examined that all the films were dense without apparent pores under scanning electron microscope (FEI Quanta 200 FEG, USA) and homogenous with surface roughness (R_a) lower than 5 nm. A scratch tester (MFT-4000), working under the conditions with a loading rate of 20 N min^{-1} , an end load of 100 N and a scratch length of 5 mm, was also employed to evaluate the adhesions between films and substrates; and the measured critical loads were higher than 30 N, indicating high-quality of the films and excellent adhesion between the titanium films and glass substrates. The thicknesses of the films measured by a three dimensional white-light interfering surface profiler (Micro XAM-3D, USA) were in the range from 170 to 2100 nm.

The nanoindentation measurements were performed with a Berkovich indenter using a fully calibrated Nano Indenter XP (Nano Instruments Innovation Center, MTS systems, TN, USA). The system has load and displacement resolutions of less than 50 nN and 0.1 nm, respectively. All the nanoindentation tests were conducted under the CSM model. At least three indentations were made in each sample and the results shown in the present contribution are the average of these measurements. All the specimens were indented at a constant loading rate, 10 mN s^{-1} , to a depth of about 2500 nm. This test procedure was performed on both the film–substrate samples and the bare glass substrate.

The Oliver–Pharr method [1] was used to calculate the hardness and reduced Young's modulus of the bare glass substrate and the film–substrate system. In calculations, the Poisson's ratios of all materials were assumed to be 0.25.

3. Results and discussion

3.1. Description of the continuous stiffness curves

Fig. 1(a) shows E_r^2/H over the penetration depth for indentation in films with different thickness. For all samples, the parameter E_r^2/H decreases with the increasing penetration depth. Similar tendency was also observed for other samples. It should be noted that, as the penetration depth increases, E_r^2/H tends rapidly to an asymptotic value for the films with smaller thickness, while a continuous decrease in E_r^2/H was observed for the films with larger thickness. This experimental phenomenon seems to be reasonable. The asymptotic value of E_r^2/H at sufficiently large penetration depth can be considered as the substrate behavior [10]. Evidently, for films with larger thickness, larger penetration depths are needed to show the asymptotic value. This can be understood more clearly by replotting Fig. 1(a) as Fig. 1(b), where E_r^2/H is shown as functions of the normalized penetration depth, h/t , where t is the film thickness. In Fig. 1(b), all the datum points for the four films fall along an asymptotic “master curve”, indicating that the differences between the curves shown in Fig. 1(a) is due to the film thickness effect. If the penetration depth were sufficiently large, the curve for the 2100-nm-thick film would also show an asymptotic value.

We now wish to perform a quantitative description for the continuous stiffness curves shown in Fig. 1. For this goal, an exponential decay function of second order, Eq. (5), was selected to fit the exper-

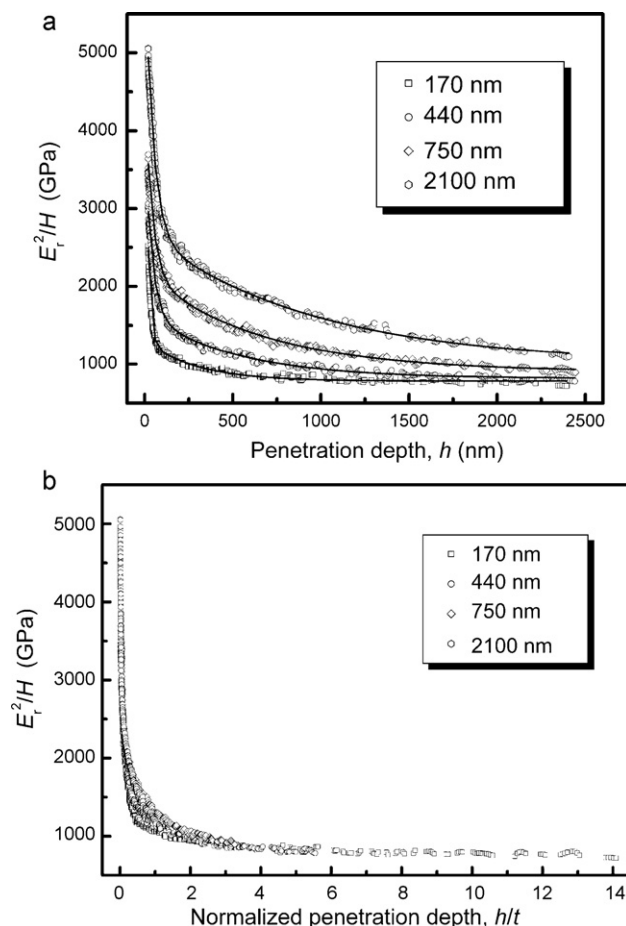


Fig. 1. E_r^2/H versus (a) indentation penetration depth and (b) the normalized indentation penetration depth for Ti films with different thicknesses on soda-lime glass substrate.

imental data shown in Fig. 1,

$$\frac{E_r^2}{H} = A_0 + A_1 \exp\left(-\frac{h}{t_1}\right) + A_2 \exp\left(-\frac{h}{t_2}\right) \quad (5)$$

where parameters A_0 , A_1 , A_2 , t_1 and t_2 are adjustable constants. Especially, t_1 and t_2 are called the exponential decay constants.

The solid lines in Fig. 1 represent the best-fit results of the experimental data according to Eq. (5) by a conventional exponential decay regression analysis. It is clear that Eq. (5) gives excellent fits to the experimental data. For the experimental data measured on other samples examined in this study, the same conclusions were also obtained.

Chen et al. [9] have measured the continuous stiffness curves for two kinds of film–substrate systems, one being Al films on glass or silicon substrate (soft film on hard substrate) and the other being W films on glass or silicon substrate (hard film on soft substrate). The reported continuous stiffness curves for both film–substrate systems are now reproduced in Fig. 2. For W films, the continuous stiffness curves are similar to those shown in Fig. 1 and Eq. (5) is proven to be suitable for describing the experimental data (solid lines in Fig. 2 are the best-fit results). For Al films, however, the behaviors are rather different from those for W films and Ti films and, clearly, the experimental cannot be described with Eq. (5). Considering that the hardness of Ti is higher than that of Al, comparison between the experimental data shown in Figs. 1 and 2 seems to indicate that there seem to be some limitations in the applicability of Equation (5). For the film/substrate systems in which the hardness of the film is much smaller than that of the substrate, Eq. (5) seems

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