Materials Science and Engineering A 488 (2008) 318–332

MATERIALS SCIENCE & ENGINEERING A

www.elsevier.com/locate/msea

Investigation on methods for dealing with pile-up errors in evaluating the mechanical properties of thin metal films at sub-micron scale on hard substrates by nanoindentation technique

Xiangyang Zhou [a,*], Zhuangde Jiang [a,b], Hairong Wang [a], Ruixia Yu [c]

[a] Institute of Precision Engineering, Xi'an Jiaotong University, Xi'an 710049, China
[b] State Key Laboratory for Manufacturing System Engineering, Xi'an Jiaotong University, Xi'an 710049, China
[c] School of Mechanical Engineering, University of Science and Technology Beijing , Beijing 100083, China

Received 7 June 2007; received in revised form 6 November 2007; accepted 7 January 2008

Abstract

Two quantitative approaches are proposed in this paper in order to deal with the significant errors produced during evaluating the mechanical properties of soft metal films on hard substrates by using nanoindention technique, which depend on calculating actual contact area (CACA: area) and correcting contact depth errors (CCDE: depth). The hardness and elastic modulus of films-only for two elastically matching systems with film thickness of about 500 nm, Au/glass-ceramic and Al/7059 glass, are then obtained by using the methods. The above parameters are also determined by the Oliver and Pharr method and the constant modulus assumption analysis (CMAA: modulus) for comparisons. In CMAA: modulus, the values are assumed to be independent of the contact area and contact depth. The results indicate that the two methods are both able to correct severe pile-up errors in the Oliver and Pharr analysis and produce a relative high accuracy.

Keywords: Thin metal films; Nanoindentation; Hardness; Elastic modulus; Pile-up

1. Introduction

Thin metal films deposited on hard substrates by various fabricating techniques, e.g. aluminum, gold or copper, etc., on silicon, glass or germanium, etc., play extremely important roles in semiconductor and microelectromechanical systems (MEMS). The reliable mechanical properties of these thin films, which are quite different from those of bulk materials, are critical to the safety and functioning of these micro-devices and should be accurately determined [1–3]. However, the thicknesses of thin films employed in these micro-devices are usually at scale levels of sub-micrometer to a few micrometers [4], so the conventional methods measuring the mechanical properties of large volume materials cannot work anymore. Therefore, a variety of testing techniques [1–5] available to small volume materials have been developed over the past decade. Among these techniques, nanoindentation has proven to be a powerful one [2] and hence is widely used to characterize the properties of thin films [6–9]. Using nanoindentation, the hardness and elastic modulus of films as thin as 1 μm-thickness can be now routinely measured [6] by the Oliver and Pharr method [10], which depends on analyzing the load–displacement data (Fig. 1) obtained from the nanoindentation tests.

However, the evaluated values by nanoindentation are normally very ambiguous because of the influences of various factors, such as the measuring device, method of evaluation and specimen [11]. The validity of the results for hardness and modulus depends largely upon the analysis procedure used to process the raw data [8]. Hence, good technique in experimental procedure and careful data analysis are required to eliminate or reduce these undesired influences. In the data analysis, particularly, one of great problems is that the Oliver and Pharr method does not consider the effect of pile-up behavior of material on evaluated properties [9]. Pile-up is often found in some ductile soft materials [11] including bulks and thin films [12–16]. The fundamental material properties affecting pile-up are the ratio of the reduced modulus to the yield stress, E_r/σ_y, and the work-hardening behavior [14]. During the indentation of thin soft metal films on hard substrates, it has been reported that pile-up is significantly enhanced due to the severe constraints

* Corresponding author. Tel.: +86 29 8266 8616; fax: +86 29 8266 8612.
E-mail address: xyz_xjtu@163.com (X.Y. Zhou).

0921-5093/$ – see front matter
doi:10.1016/j.msea.2008.01.020

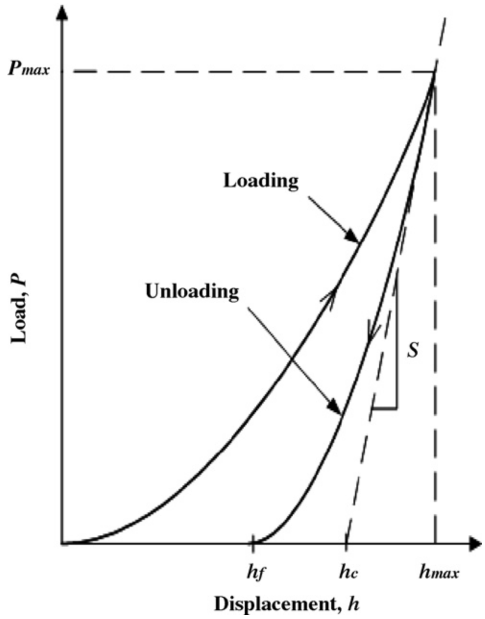

Fig. 1. Schematic illustration of a typical indentation load–displacement curve generated during one complete cycle of loading and unloading.

imposed by the hard substrate, which impede the downward flow of the film material and thus result in the material tending to plastically pile-up around the sides of the indenter. In some cases, it has a much greater degree than in the case of monolithic materials [1,12,13,16], as shown in Fig. 2. For a specific film thickness, due to the presence of hard substrate the extent of pile-up increases monotonically with increasing indentation depth [1,9], which might be associated with strain gradient effects [13,15]. When it occurs, the Oliver and Pharr method would significantly underestimate the contact area and hence overestimate the hardness and elastic modulus [14–16], especially when the indenter tip approaches the film–substrate interface [15]. Therefore, the pile-up errors must be corrected in the data analysis if reliable hardness and elastic modulus of thin metal films are to be obtained. However, it is often difficult because the indentation response is a complex function of not only the plastic but also elastic properties of both the film and the substrate [13], so the influences of the mismatch in properties of the film and substrate on measured results need to be simultaneously taken into account during dealing with the pile-up errors. For a soft film on hard substrate system that the substrate has higher modulus than the film, particularly, due to the influence of substrate property, it is observed that the hardness and elastic modulus of thin film predicted by the Oliver and Pharr method increase gradually with indentation depth increasing [16,17], indicating an artifact in evaluated results that obscures the intrinsic properties of thin film.

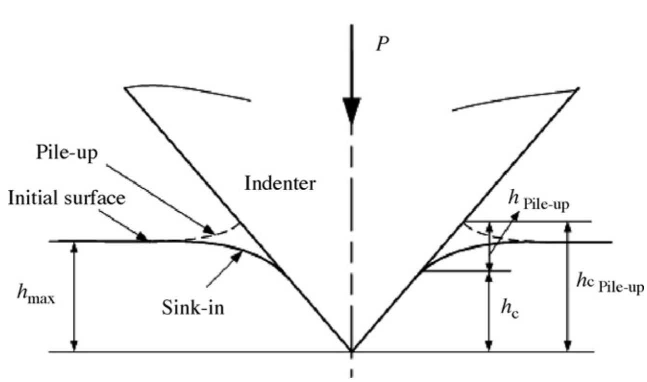

Fig. 2. Schematic illustration of cross-sectional profile for a conical indentation showing the pile-up/sink-in behavior.

To determine the 'true' properties of thin film without the substrate effects, a commonly used rule of thumb is that the indentation depth is limited to less than 10% of the film thickness [9,12]. While this rule is experimentally feasible for films that are greater than about 1 μm in thickness, it cannot be used for very thin films [16] due to the severe indentation size effect (ISE) [18]. For this situation, the careful data analysis is specially required. In many previous investigations, various researchers have attempted to extract the 'absolute' hardness and elastic modulus of thin films from the composite results combined the both substrate and film contributions by using experimental [9,12,13,16,17,19] and theoretical [1,20–26] methods.

In this paper, we make our effort to investigate the methods to deal with the pile-up errors during the nanoindentation measurements of the soft metal films at sub-micron scale on hard substrates. Based on the Oliver and Pharr analysis for the case of a perfect Berkovich diamond tip indentation, methods to correct the pile-up errors in the determination of hardness and elastic modulus are theoretically analyzed and the corresponding formulas are derived; they are available for the cases of monolithic materials and elastically matching film–substrate systems. Since the film and the substrate have very similar elastic moduli, the systems are nearly elastically homogeneous and hence the influence of the elastic mismatch between thin film and substrate on indentation response could be minimized [12]. And then, the methods are applied to two elastically matching systems with film thickness of about 500 nm, i.e., Au/glass-ceramic and Al/7059 glass. Furthermore, the results are compared with those determined by using the Oliver and Pharr method and the constant modulus assumption analysis [16,17,27].

2. Analytical background

In the Oliver–Pharr analysis [10], the hardness H and elastic modulus E are determined from the load–displacement curve (Fig. 1). The hardness is defined as

$$H = \frac{P_{\max}}{A} \tag{1}$$

where $P_{\max}$ is the maximum indentation load and A is the projected contact area at $P_{\max}$.

The reduced elastic modulus E_r is derived from the relation:

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \tag{2}$$

where S is the contact stiffness computed from the initial slope of the unloading curve at the $P_{\max}$. β is a constant that depends on the geometry of the indenter ($\beta = 1.034$ for a Berkovich indenter