Thin Solid Films 544 (2013) 201–205

Contents lists available at ScienceDirect

Thin Solid Films

journal homepage: www.elsevier.com/locate/tsf

Determination of Young's modulus and Poisson's ratio of thin films by X-ray methods

Wei-En Fu *, Yong-Qing Chang, Bo-Ching He, Chung-Lin Wu

Centers for Measurement Standards, Industrial Technology Research Institute, No.321, Sec.2, Kuangfu Rd., Hsinchu 30011, Taiwan

ARTICLE INFO

Available online 10 April 2013

Keywords:
Thin film
X-ray diffraction
Young's modulus
Poisson's ratio
Copper
Residual stress

ABSTRACT

Accurate determination of Young's modulus and Poisson's ratio is critical when characterizing the mechanical properties of ultra-thin films. In this work, a method to simultaneously measure the Young's modulus and Poisson's ratio of thin films by X-ray techniques combined with a four-point bending fixture is presented. The four-point bending fixture was carried out to purposely induce stresses on the thin film and cause a curvature of the film/wafer system. The induced stresses were measured by the modified conventional X-ray diffraction for film measurements. At the same time, the curvature of the film/wafer system was measured by the X-ray rocking curve. With the film stress derived from the curvature according to the Stoney formula, both the Poisson's ratio and the Young's modulus were evaluated from the plot of lattice *d*-spacing changes (strain) versus $\cos^2\alpha \sin^2\psi$. The method was applied to determine thin copper films (~180 nm). The estimated Poisson's ratios ranged from 0.18 to 0.24. Compared to the 0.34 for bulk Cu, the deviation between the bulk and the film properties was above ~30%. The Young's modulus of the films is 116.7 ± 2.9 GPa, which was comparable to those reported in the literature for ~128 GPa.

1. Introduction

The mechanical properties of thin films are of great importance in many engineering applications such as integrated circuits and micro-electro-mechanical systems, since they are directly related to device reliability [1]. Mechanical properties, such as residual stress, play an important role in thin films, since it could be detrimental to the film system if the existing stress is tensile and leads to crack initiations or pattern line deformation during or after subsequent manufacturing processing [2,3]. Two methods are frequently used to measure the residual stresses in the analysis and measurements in thin films. One is bending beam method to calculate the residual stress in thin films globally by applying the Stoney formula on a film/wafer system [4,5]. The residual stress in the film on a wafer substrate causes the film/wafer system to curve until mechanical equilibrium is reached. Without knowing the elastic properties of the films, the residual stress can be determined from the easily obtained curvature of the bent film/wafer system. The other one is the X-ray diffraction (XRD) method to decide the localized stress, where Bragg's law is applied to decide the elastic strain variations for stress calculations using Hooke's law [6–9].

When the residual stress is converted from stress–strain relationship based on Hooke's law in the XRD measurements, it requires both known elastic constant and Poisson's ratio, which are generally estimated from the bulk materials [6,10]. However, when films are getting thinner to meet the demands of semiconductor industries, the film properties, such as density and structures, are not the same as the bulk materials [11]. The use of elastic constant and Poisson's ratio for bulk materials will cause erroneous residual stress measurements by the XRD method.

Several methods were proposed to measure the elastic properties of thin films combining XRD method and bending beam method [11–16]. The XRD method measures the out-of-plane strain, and the bending beam method measures the average in-plane stress of the film [17,18]. With the measured out-of-plane strain and in-plane stress, the Young's modulus and the Poisson's ratio of thin films can be derived from the shear modulus G where $G = 2E / (1 + \upsilon)$. Laser scanning was the most frequently used in the bending beam method to measure in-plane stress from the curvatures, where the curvatures of the bending beam were frequently obtained from heating. Such combination proved to be effective as a non-destructive method to measure the Young's modulus and the Poisson's ratio promisingly for thin films [12,14,16,17,19].

While the combination method is promising for measuring the Young's modulus and the Poisson's ratio, it also has difficulties to incorporate laser scanning into XRD in one setup to measure the out-plane strain and in-plane stress at the same time. In this article, however, the X-ray rocking curve (XRC) measurement was used to replace laser scanning to measure the curvatures of the films. In this setup, we were be able to measure the out-plane strain by the XRD and in-plane stress by the XRC in the same setup and at the same time. A four-point bending fixture was applied to bend the film/wafer system and provide the curvatures of the films. The combination of

---

* Corresponding author. Tel.: +886 3 5732220; fax: +886 3 5726445.
E-mail address: WeienFu@itri.org.tw (W.-E. Fu).

0040-6090/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.tsf.2013.03.121

XRD and XRC method was applied to estimate the elastic modulus and the Poisson's ratio of thin Cu films with thickness of ~180 nm.

2. Experiments

2.1. Sample preparation and characterization

A single layer of Cu was deposited on Si (1 0 0) wafers by using commercial ion beam-assisted deposition (IBAD) at the room temperature. The vacuum chamber was evacuated to 7×10^{-5} Pa prior to deposition. The deposition was processed under bias 800 V and current 8 mA with Ar mass flow of 3 sccm at working pressure ~ 4×10^{-2} Pa. The thickness of the Cu films was ~180 nm, which was determined by X-ray reflectivity (XRR) based on a material structure model of Si/SiO_2/Cu/Air, as shown in Fig. 1.

2.2. X-ray techniques

In order to generate curvatures and stresses to the Cu films, a piezoelectric ceramic (PZT) multilayer stack-type actuator was buried levelly in the middle of a four-point bending fixture. The thin Cu films were then mounted on a four-point bending fixture above the actuator. The external stress applied to the Cu films came from the actuator with varied input voltages of 50 V, 75 V and 100 V. The overall apparatus of the four-point bending fixture is shown in Fig. 2.

The stress produced in the film based on the four-point bending fixture can be estimated by the following equation [20]:

$$\sigma_f = E_f \times \frac{M}{E\cdot I}\left(h_s + \frac{h_f}{2} - \frac{E_f\left(2h_s\cdot h_f + h_f^2\right) + E_s\cdot h_s^2}{E_s\cdot h_s + E_f\cdot h_f}\right) \qquad (1)$$

where the M is the bending moment applied to the film and substrate, E_f is the elastic modulus of the film, E_s is the elastic modulus of the film, h_f is the thickness of the film, h_s is the thickness of the substrate, and $E \cdot I$ is the stiffness of the composite of film and substrate. A piezoactuator was used to provide a force F to generate the bending moment M, as:

$$M = F \times a \qquad (2)$$

where the F is the force applied from the piezoactuator underneath the substrate through the middle two pins, as shown in Fig. 2, and a is the distance between one of the middle pins to its adjacent supporting pin. Before measurements, a pre-loading (the input voltage was ~5 V) was applied to make sure of the contact of the piezoactuator and the bottom of the substrate. Subsequently, the 50 V, 75 V and 100 V input voltages were applied to produce bending moments for creating film stresses and curvatures. Since the measurements by XRD and XRC can be performed simultaneously in the same setup, the 'same' stresses and curvatures are obtained.

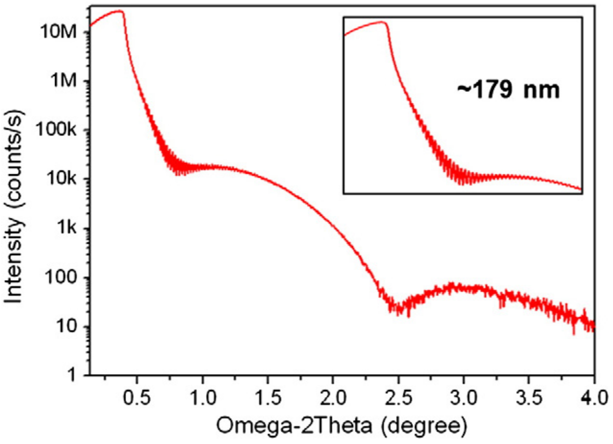

Fig. 1. As-deposited Cu film thickness of ~180 nm measured by XRR based on a sample model of Si/SiO_2/Cu/air.

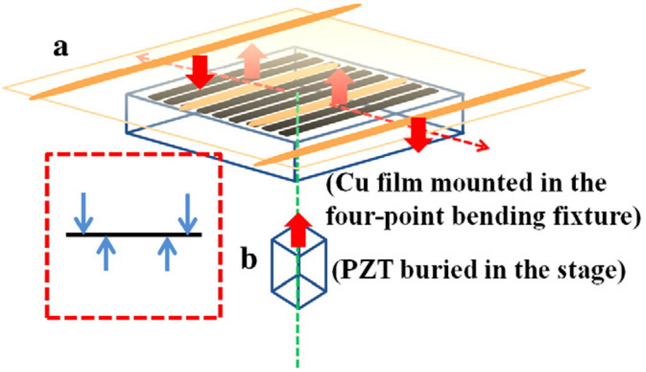

Fig. 2. (a) Four-point bending fixture, combined with (b) PZT multilayer stack-type actuator to produce external stress for the Cu films.

X-ray reflectivity, asymmetric XRD diffraction and the curvature measurements were all performed on a commercial XRD diffractometer (PANalytical X'Pert PRO MRD). A metal-ceramic Cu-target X-ray tube at 45 kV and 40 mA was used for the generation of X-rays. The incident and diffracted X-ray beams were conditioned using an X-ray mirror and a parallel plate collimator. The samples were mounted on a four-circle goniometer, where the film tilted angle ψ was at −80°, −60°, −40°, −20°, 0°, 10°, 20° and 30°. For the asymmetric XRD diffraction configuration [8,9], a grazing angle of X-ray incidence (point focus) was set at 0.5° and the detector scanning in the 2θ ranges from 41° to 45° for the characteristic peak ($2\theta = 43.473°$, JCPDS #00-001-1241) of Cu (1 1 1). For the film/wafer curvature measurements, the XRC (line focus) was applied for observing the shifts of the Si (1 0 0) characteristic peak of $2\theta = 69.583°$ (JCPDS #00-003-0517) with input voltages at 50 V, 75 V and 100 V.

3. Results and discussion

3.1. Theoretical background

The traditional XRD method for residual stress measurement is known as the $\sin^2\psi$ method, which is based on the measurement of the shift of a diffraction peak position recorded for different ψ angles based on θ–2θ rotation [16,21]. In this conventional Bragg–Brentano (B–B) geometry approach, a specific diffraction plane is selected and the interplanar spacing is measured from a coupled θ–2θ scan of the specimen at different tilt angle ψ (the angle between the diffracting plane normal and the specimen surface normal) [10]. However, commonly for the films, which are highly textured or single crystalline-like, under the symmetric B–B diffraction geometry, only a few specific (h k l) peaks can be found. Especially in consideration of the diffraction volume in thin films, high angle diffraction peaks might be too weak to be conveniently measured even if they are presented [7].

In this experiment, asymmetric XRD diffraction was applied to maximize the diffraction volume of thin films [8], as shown in Fig. 3. Unlike the traditional $\sin^2\psi$ method, the asymmetric XRD diffraction proposed an "asymmetric" B–B XRD geometry [7,8], with the incident X-ray beam at a grazing angle ω with respect to the specimen surface [8]. The grazing incident angle in this configuration of the XRD was $\omega = 0.5°$. The (1 1 1) characteristic peak was kept constant with different ψ tilt angles for the asymmetric diffraction configuration. Thin film strain measured using the asymmetric XRD diffraction geometry was assumed a linear relationship versus $\cos^2\alpha \sin^2\psi$, where the angle α is