Rare Metal Materials and Engineering
Volume 45, Issue 4, April 2016
Online English edition of the Chinese language journal

Available online at www.sciencedirect.com
ScienceDirect

Cite this article as: Rare Metal Materials and Engineering, 2016, 45(4): 0843-0848.

ARTICLE

Effect of Different Substrate Temperatures on Microstructure and Residual Stress of Ti Films

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Abstract: The influence of different substrate temperatures of Ti film prepared by the direct current (DC) magnetron sputtering on the internal residual stress of Ti film were investigated experimentally by nanoindentation technique including Suresh model and Lee model. The results of the nanoindentation method and the curvature method were compared. At the same time, the surface morphology and the microstructure of Ti film were analyzed using atomic force microscope (AFM) and X-ray diffraction (XRD). The results show that the residual stress value obtained by Suresh model is almost the same as that by the curvature method, so the Suresh model is more suitable for calculating the residual stress of Ti films. Together with the nanoindentation data and micro-structure analysis, it is found that with the substrate temperature rising, the grain size of Ti films increases first and then decreases; the residual stress of the Ti film changes from the compressive stress to the tensile stress.

Key words: nanoindentation; curvature method; residual stress; film

With the development of optoelectronic technology, the applications of thin films to optics and electronic devices are found everywhere. However, residual stress exists in almost all films, due to the lattice mismatch or different thermal expansion coefficients between the film and the substrate[1]. The residual stress will impact the structure and the service performance of the film, and lead to crack formation and fatigue in the film or make the film become invalid under the circumstances, which greatly shortens the service life of the product[2]. Titanium and its alloys are important materials in the fields of modern life science, military and aerospace. They have high strength and favorable corrosion resistance performance, as well as the advantages of nice biological compatibility[3]. Therefore, it is very important to research the residual stress in DC magnetron sputtering Ti film.

Currently, many researchers have measured the residual stresses of thin films employing different methods such as X-ray diffraction method, curvature method and Raman spectroscopy technique. X-ray diffraction method is only applied to measure the lattice semiconductor or crystal film, and has certain requirements on the film thickness [4]. Raman spectroscopy technique is often used to measure the residual stress of MEMS (micro-electro-mechanical system) devices, but this method is some restricted by fluorescence interference and low detection sensitivity [5, 6]. Curvature method has characteristics of convenience and high accuracy which can obtain the average residual stress without destroying samples. It's widely used in the measurement of thin film residual stress, but this method has a high requirement of the roughness on the surface of the sample. In place of these methods, the present study focuses on a technique to evaluate residual stress using an indentation method.

Residual stress measurement by the nanoindentation technique has been considered a promising process. Nanoindentation is a method which uses the displacement and the strain that result from the partial load to infer the

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Received date: April 20, 2015
Foundation item: National Natural Science Foundation of Beijing (3120001); National Natural Science Foundation of China (51275105); Distinguished Young Scholars of National Natural Science Foundation of China (51125023)

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residual stress of the material. In the present paper, the residual stresses in the Ti films at different substrate temperatures were investigated by nanoindentation. The residual stresses were calculated based on the theoretical models of Suresh et al. [7] and Lee et al.[8]. By comparing with the results of curvature method, the error sources were analyzed.

1 Theory of Two Residual Stress Measurement Methods

1.1 Theory of nanoindentation

At present, nanoindentation techniques are widely used for measuring the mechanical properties of both solids and thin films, and allow the measurement of residual stress at nanoscale [9]. Several methodologies have been developed for the determination of the residual stresses from the indentation *P-h* (load-depth) curves. Suresh model and Lee model determined biaxial residual stresses using sharp indentation Swadener model [10] based on spherical indentation. The contact pressure at initial yielding was proposed as a stress indicator via analyzing the elastic deformation beneath the spherical indenter. Recently, with the application of finite element simulations, Xu and Li et al [11,12] have systematically investigated the influence of residual stress on the elastic recovery of nanoindentation. Suresh model and Lee model are widely used amongst them. Although the principles of measurement are not identical, Lee model and Suresh model are applicable to biaxial residual stress state. In the present article, the residual stress was calculated by the model of Suresh and Lee to determine which one was more suitable for measuring the residual stress of Ti films.

In 1998, a theoretical model using a geometrically similar sharp indenter was proposed by Suresh and Giannakopoulos[7] to characterize the equi-biaxial thin-film stress. In this model, the residual stress in the film is determined based on the indentation contact area difference between a residual stress-free material and the same material with residual stress. The residual stress σ^{R} can be calculated by the following equations.

For tensile residual stress:

$$\sigma^{\mathrm{R}} = H\left(\frac{A_0}{A} - 1\right) \tag{1}$$

For compressive residual stress:

$$\sigma^{\mathrm{R}} = \frac{H}{\sin\alpha}\left(1 - \frac{A_0}{A}\right) \tag{2}$$

where H is the hardness of the material. $\sin\alpha$ is a geometric factor, where α is related to the indentation angle of the indenter. For a Berkovich indenter, α=24.7° and $\sin\alpha$= 0.418. A and A_0 are the projected contact areas for materials with and without residual stress σ^{R}, respectively.

In 2004, Lee and Kwon[8] developed a new method to estimate two-dimension residual stress based on the analysis of the stress relaxation during the depth-controlled indentation. The model can measure the residual surface stress in the equi-biaxial and uniaxial states. Two major stresses are σ_x and σ_y ($\sigma_y = k\sigma_x$), and σ_x can be calculated by the following equation:

$$\sigma_x = \frac{3(P - P_0)}{(1 + k)A_1} \tag{3}$$

where P and P_0 are the maximum loads for the materials with and without residual stresses, respectively. A_1 is the projected contact area for the material with stress state. For the state of equi-biaxial residual stress, k =1[13].

1.2 Theory of curvature method

Curvature method is suitable for calculation of two-dimensional stress state, which has the same premise as nanoindentation models. It is assumed that isotropic biaxial stress exists in the film which is at the same temperature. Substrate will bend slightly when the two-dimensional residual stress appears after the film is formed. The residual stress is determined by measuring the change of the substrate curvature before and after film deposition. The parallel monochromatic light is used by electronic film stress distribution tester to form the interference between the surfaces of flat plane and film. The residual stress is determined by measuring the change of the interference fringe before and after film deposition. The residual stress can be calculated by the Stoney equation [14]:

$$\sigma = \frac{E_{\mathrm{s}}}{1 - \nu_{\mathrm{s}}} \cdot \frac{t_{\mathrm{s}}^2}{6t_{\mathrm{f}}} \cdot \left[\frac{1}{R_1} - \frac{1}{R_2}\right] \tag{4}$$

where σ is residual stress in the film, t_{s} and t_{f} are the thicknesses of the substrate and thin film, respectively. E_{s} and ν_{s} are the young's modulus and Poisson's ratio of substrate, respectively. R_1 and R_2 are the curvatures of the substrates before and after the film deposition, respectively. This method requires the shapes of the substrate be circular or rectangular[15].

2 Experiment

2.1 Specimen preparation

An isotropic Ti film was deposited on (100) single side polished Si wafers with diameter of 50 mm in order to exclude the additive effects of microstructure and anisotropic deformation on the nanoindentation curve. The films were made by the AS500DMTXB automatic computer-control ion evaporator. The purity of Ti target was 99.99%. The substrate was dipped into acetone and cleaned with ultrasonic washer for 15 min, and then it was dried in vacuum for deposition. The substrate was cleaned for 3 min by argon with the washing flow of 200 cm^3/min and bias voltage of 100 V. The reaction vacuum degree and power were 0.88 Pa and 0.5 kW, respectively. Ti thin films with

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