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# Evaluation of residual stress in sputtered tantalum thin-film

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ABSTRACT

The influence of deposition conditions on the residual stress of sputtered tantalum thin-film has been evaluated in the present study. Films have been deposited by DC magnetron sputtering and curvature measurement method has been employed to calculate the residual stress of the films. Transitions of tantalum film stress from compressive to tensile state have been observed as the sputtering pressure increases. Also, the effect of annealing process at temperature range of 90–300 °C in oxygen ambient on the residual stress of the films has been studied. The results demonstrate that the residual stress of the films that have been deposited at lower sputtering pressure has become more compressive when annealed at 300 °C. Furthermore, the impact of exposure to atmospheric ambient on the tantalum film stress has been investigated by monitoring the variation of the residual stress of both annealed and unannealed films over time. The as-deposited films have been exposed to pure Argon energy bombardment and as result, a high compressive stress has been developed in the films.

## 1. Introduction

Tantalum (Ta), a refractory metal, has a wide range of applications in a variety of fields. Due to its high melting point (3290 K), high density, high fracture toughness and corrosion resistance, tantalum is a promising material in bio-medical, micro electromechanical system (MEMS) devices and high-temperature applications [1]. Tantalum thin-films have been playing an important role in electronic devices acting as a diffusion barrier between metals and silicon substrate [2–4], as an alternative of hazardous electrodeposited chromium coatings [5] and an excellent x-ray mask absorber [6]. Tantalum is used also as a hardener coater on titanium surface for orthopaedic implants applications [7] and to protect steel and glass substances from wear and corrosion [8,9]. Recently, tantalum has been employed successfully for fabricating buckled and straight MEMS beams [10] and as a metal bridge structure for the resonant gate transistors (RGTs) that could be vibrated at low range of frequency suitable for audio applications [11].

However, one of the main obstacles that may lead to functional failure in a device is the residual stress of the material. For instance, buckling and cracking in films, wafer curvature and changes in device functions could be attributed to undesirable stress effects [12,13]. Despite numerous attempts to characterise the stress in thin-film materials, the real mechanism is not well understood. Several approaches have been reported to control the residual stress or improve the physical properties of tantalum thin-film for a specific application. It has been found that the residual stress and crystallographic structure of tantalum thin-film can be manipulated in various ways, such as annealing at different temperatures, using different substrates and deposition techniques and changing the deposition conditions. Previous work have been published about the influence of annealing process, thermal cycling and oxygen diffusion on the intrinsic stress and phase transition between alpha ($\alpha$) and beta ($\beta$) of tantalum thin-films [14–21]. The stresses of tantalum films that have been deposited on different substrates including silicon [14,15], silicon dioxide [14,17], stainless steel [5,22], glass [13,23], and titanium [24] have been investigated. Several studies have been reported on the stress of tantalum thin-films as a function of substrate bias voltage [6,22], deposition system [6,20,22,23,25], deposition conditions [10,20,23,26–29], substrate temperature [6], ion bombardment [30] and film thickness [5,12].

However, previous studies have limited their research on a particular aspect of deposition or annealing conditions and the conditions' influence on the phase transformation of the tantalum films. In our study, we aim to provide a comprehensive investigation of the tantalum film stress as a function of deposition conditions, annealing treatment, exposure of annealed and unannealed film to atmospheric ambient and ion bombardment exposure. The present work utilises the curvature method to deter-

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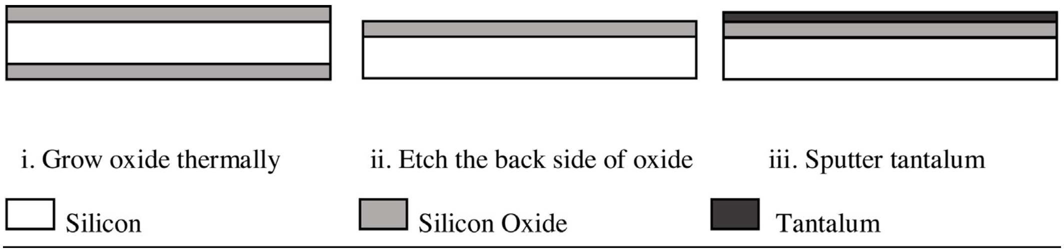

**Fig. 1.** Fabrication process flow for tantalum thin-film.

**Table 1**
Sputtering parameters of tantalum thin-films in DC magnetron system using argon (Ar) as sputtering gas with flow rate of 50 sccm.

| Wafer | Power (W) | Pressure (mTorr) |
|---|---|---|
| 1 | 300 | 3.5 |
| 2 | 300 | 7.5 |
| 3 | 300 | 11.5 |
| 4 | 300 | 19 |
| 5 | 300 | 24 |
| 6 | 400 | 3.5 |
| 7 | 400 | 7.5 |
| 8 | 400 | 11.5 |
| 9 | 400 | 19 |
| 10 | 400 | 24 |
| 11 | 500 | 3.5 |
| 12 | 500 | 7.5 |
| 13 | 500 | 11.5 |
| 14 | 500 | 19 |
| 15 | 500 | 24 |

mine the total residual stress, including: intrinsic stress that arises from the microstructure and deposition process, thermal stress that occurs due to the thermal coefficient differences, and extrinsic stress that arises from post-deposition processes. Importantly, the main objective of this study is to identify the conditions of sputtering, annealing and air or ion bombardment exposures under which the residual stress will behave in a more compressive or tensile manner. The anticipated outcomes of these results are very valuable and could be employed in the fabrication of MEMS devices for sensing and actuation applications.

## 2. Experiment details

Fig. 1 shows the fabrication steps of tantalum thin-film. To begin with, about 0.5 μm thick oxide has been grown thermally on both sides of a silicon substrate (4-inch, <100> p-type). After removing the backside of oxide via reactive ion etching (RIE), an initial set of curvature measurement has been performed using (Veeco Dektak 8000 Surface Profiler). Then, a set of 50 nm tantalum thin-films have been deposited on the top oxide by DC magnetron sputtering system (OPT Plasmalab system 4000). The sputtering conditions are presented in Table 1.

In this process, a step has been created which enables film thickness to be measured by atomic force microscopy (AFM) and Dektak profilometer. Afterwards, a second set of curvature measurement has been carried out and the residual stress $\left(\sigma^f\right)$ of the films has been estimated based on Stoney's formula:

$$\sigma^f = \frac{E_s T_s^2}{6(1-\upsilon_s)T_f}\left(\frac{1}{R_{s+f}} - \frac{1}{R_0}\right) \qquad (1)$$

where $E_s$ is the modulus of the substrate (MPa), $\upsilon_s$ is Poisson's ratio of the substrate, $T_s$ is the substrate thickness (μm), $T_f$ is the film thickness (μm), $R_0$ is the initial substrate radius of curvature (μm) and $R_{s+f}$ is the final radius of curvature (μm) of substrate plus the thin-film.

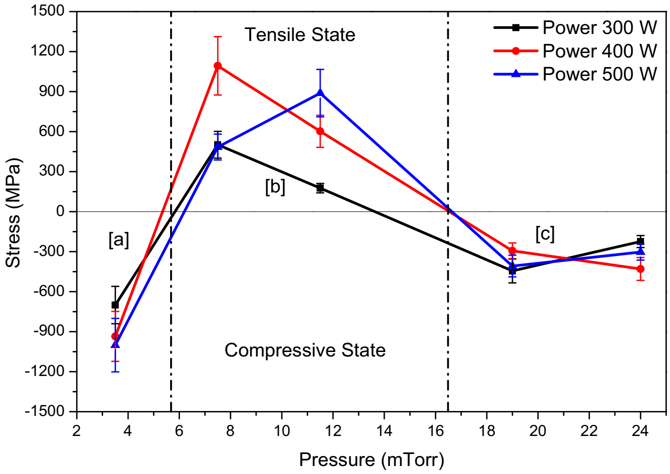

**Fig. 2.** The residual stress of tantalum films as a function of sputtering pressure for different sputtering powers.

## 3. Results and discussion

### 3.1. Effect of sputtering pressure and power

The residual stress measurements of as-deposited tantalum films as a function of sputtering pressure for powers (300, 400 and 500 W) are shown in Fig. 2. Three main regions of the residual stress in the films are shown in the figure: compressive stress (regions a and c) and tensile stress (region b). It can be seen that the effect of sputtering power on the degree of residual stress in the films have been found to be significantly larger in the tensile state region. In the compressive stress regions, increasing the power has produced a slight change in the residual stress of the films. Consistent with literature findings [10,20,26,27,31], the residual stress begins in a compressive state (region a) at low pressure (3.5 mTorr) and as the sputtering pressure increases, the trend of stress switches to a tensile state (region b) at 7.5 and 11.5 mTorr before shifting back again to the compressive state (region c) at 19 and 24 mTorr respectively.

The compressive stress exhibited in tantalum films at low sputtering pressure (region a) could be because of the atomic peening. The atomic peening mechanism has been described in more detail elsewhere [23,28,32]. In such a scenario, the argon and target atoms have long mean free path and high momentum with fewer collisions, thus bringing about a compressive stress in the film. Therefore, the compressive-stressed film at low sputtering pressure might have dense fibrous structures as described by Yoshihara and Suzuki [33].

In the second region (b), the stress of tantalum films is observed to change from compressive to tensile around 7.5 mTorr. It seems that the tensile-stressed films could possess ultra-small columnar grains with low density grain boundaries at the film/substrate interface [20], and this abrupt transition could be attributed to constrained grain boundary relaxation [23,28,32]. In