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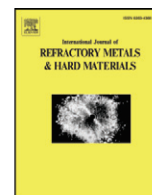

The effect of vacuum annealing on the microstructure, mechanical and electrical properties of tantalum films

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ABSTRACT

The microstructure, mechanical and electrical properties of vacuum annealed tantalum films were studied. X-ray diffraction spectra confirmed the presence of mixed (α and β) phases in the as-deposited Ta films. After vacuum annealing (at 750 °C for 1 h), the metastable β-phase was completely transformed to stable α-phase. The grain size increased (from 35 ± 3 nm to 92 ± 3 nm) with the increase in annealing temperature. The mixed (α and β) phases resulted in higher hardness and higher Young's modulus. The film annealed at 750 °C for 1 h exhibited lower resistivity (52 ± 4 μΩ-cm), lower hardness (H = 10.4 ± 1.3 GPa) and lower Young's modulus (Y = 185 ± 5 GPa) as compared to the as-deposited and annealed (at temperature < 750 °C) films. This is attributed to the phase transformation from β to α at an annealing temperature of 750 °C.

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1. Introduction

Tantalum (Ta) is one of the refractory metals which in thin film form is of interest for applications in microelectronic devices such as resistors, heaters and capacitors [1–4]. Due to high melting temperature (~3050 °C), high strength, and good corrosion and wear resistance, such electronic devices can work in harsh environment and/or at high temperatures [5]. Bulk Ta has a body-centered cubic (bcc) structure but in thin film form it grows into two crystalline structures; bcc structure (known as α-phase) and a metastable tetragonal structure (also known as β-phase) [6,7]. Both crystalline phases exhibit different electrical and mechanical properties, thus defining different areas of applications. For example, due to low resistivity (15–70 μΩ-cm) and low hardness (~8–12 GPa), the α-phase Ta is suitable for use as a diffusion barrier in integrated circuits (ICs) to stop copper diffusion [8]. The β-phase Ta exhibits high resistivity (150–210 μΩ-cm) and high hardness (~18–20 GPa), thus suitable for being use in fabricating the resisters and capacitors [9].

Various groups grew Ta films either by electron beam evaporation [10,11] and/or sputtering [3,4,12,13] to study the effect of deposition parameters [11,13–15] and substrate material [13] on the phase formation in as-deposited Ta films. Further, the effect of substrate temperature [10] and annealing conditions [16] on the structure, mechanical and electrical properties of Ta films has also been studied. For example, Liu et al. [16] reported that the Ta films (grown on Si substrate) annealed at 750 °C (for 30 min) under low vacuum (2.7×10^{-2} mbar) resulted in the formation of mixed phases (α and β) of Ta along with a Ta_2O_5 phase. Under low vacuum annealing, the Ta films were oxidized due to the presence of some oxygen content and/or moisture inside the chamber/furnace [17]. Dorranian et al. [18] while studying the effect of substrate temperature observed only β(410) peak in Ta films. Arshi et al. [10] studied the effect of substrate temperature on the structure of Ta films and observed β(330) peak in all films. Though the texture was different in both of these studies, the Ta films grown on either glass or Si/SiO_2 substrates exhibited a β-phase. It is known that the metastable β-phase transforms to stable α-phase during post-annealing at temperatures ranging from 600 to 800 °C [19]. Further, in most of the reports on vacuum annealed Ta films either the phase transformation or film stresses are studied but according to our knowledge the mechanical properties in vacuum annealed Ta films are rarely studied [16,17,19,20]. In vacuum annealed Ta films, the phase transformation depends on the annealing parameters such as vacuum conditions, annealing temperature, annealing time and substrate nature. Further, it has been established in Ref. [13] that the as-deposited Ta films exhibit a single α-phase if the films of thickness, $t \leq 200$ nm were grown on Si(100) substrates at low (≤6.0 mTorr) sputter pressure.

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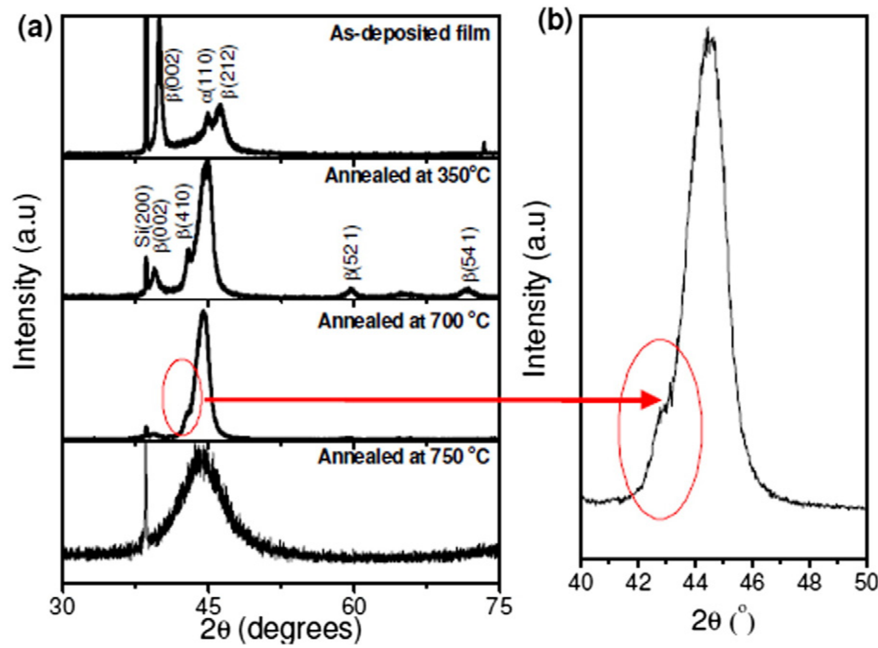

Fig. 1. (a) X-ray diffractograms of as-deposited and annealed Ta films at different temperatures for 1 h. (b) Extended XRD pattern (for 2θ = 40°–50°) of vacuum annealed (at 700 °C for 1 h) tantalum film, where asymmetrical α(110) peak indicates the presence of minor fraction of β-phase.

In this communication, we report some results studying the effect of vacuum annealing on the microstructure, mechanical and electrical properties in Ta films of fixed thickness.

2. Experimental detail

Tantalum films were grown on Si(100) substrates using a Kurt J. Lesker DC magnetron sputtering system. The films thickness (500 ± 2 nm) was chosen on the basis of our previous study [13]. The substrates were washed (for 20 min) in an ultrasonic bath using acetone and ethanol to remove any contamination from substrate surface. The washed substrates after drying with nitrogen gas were immediately placed into the sputter chamber. Before deposition, the achieved base pressure of the chamber was better than 2.7×10^{-6} mbar. Tantalum target (purity 99.99%) of 2 mm in thickness and 50.8 mm in diameter was used for sputtering. The sputter parameters such as sputter power (100 W), argon sputter pressure (7.3×10^{-3} mbar) and the substrate-to-target distance ($d = 7.5$ cm) were kept constant during the growth of all films. The films were grown without applying any bias voltage across the substrate. After deposition, the films were annealed under vacuum using a vacuum furnace with a rotary/diffusion pump combined outfit. Before switching on the furnace, a vacuum of 6.7×10^{-6} mbar was achieved. During annealing, the temperature was increased to the set point temperature at the rate of 8 °C/min. After maintaining the set point temperature for 1 h, the film was then allowed to cool down to room temperature under vacuum to avoid any oxidation.

The crystal structures of all films were studied by X-ray diffraction (XRD). A Siemens D5000 X-ray diffractometer with Co-radiation (wavelength, λ = 1.78896 Å) operating at 30 kV and 40 mA was employed. An X-ray diffractometer was run in the Bragg–Brentano (θ–2θ) geometry to collect XRD data of all films. During XRD measurements, the sample was rotated about its own axis, so that the incident X-ray beam could diffract from all orientations of the crystallites. An atomic force microscope (AFM; IIIa Veeco Digital Nanoscope) was used (in tapping mode) to elucidate the surface morphology, grain size and surface roughness. Nanoindentation measurements were taken using an AFM (Veeco Dimension 3100 Nanoscope) with a Berkovich diamond indenter. For each film, at least eight measurements were taken to collect the load–displacement data by setting the indentation depth not more than 20% of the film thickness. This allows eliminating the substrate effect during indentation measurements. From load–displacement data, the average hardness, H and Young's modulus, Y were calculated by adopting the procedure proposed by Oliver and Pharr [21]. The resistivity of all films was measured by a four-point-probe method as described in Ref. [13].

3. Results and discussion

3.1. Microstructure and phase analysis

Fig. 1a shows the XRD diffractograms of as-deposited film and vacuum annealed Ta films. The as-deposited film (Fig. 1a) exhibits two peaks (002) and (212) corresponding to the β-phase (JCPDS-25-1280) while a low intensity (110) peak corresponds to the α-phase (JCPDS-04-0788).

Table 1
Summary of structural parameters for vacuum annealed tantalum films.

Annealing temperature (°C)	Phase	Fraction of β-phase f_β (%)	Lattice constant of α-Ta a_α (Å)	Lattice constant of β-Ta (in Å)	c_β/a_β
As-deposited	$\alpha + \beta$	82.2	3.304 ± 0.025	$a_\beta = 10.437 \pm 0.495$ $c_\beta = 5.236 \pm 0.009$	0.502
350 °C	$\alpha + \beta$	36.8	3.326 ± 0.001	$a_\beta = 10.200 \pm 0.048$ $c_\beta = 5.293 \pm 0.004$	0.519
700 °C	$\alpha + \beta$	5	3.347 ± 0.001	–	–
750 °C	α	0	3.355 ± 0.003	–	–

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