Surface & Coatings Technology xxx (2016) xxx–xxx

Contents lists available at ScienceDirect

Surface & Coatings Technology

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

Phase tailoring of tantalum thin films deposited in deep oscillation magnetron sputtering mode

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ARTICLE INFO

Article history:
Received 30 June 2016
Revised 4 August 2016
Accepted in revised form 6 August 2016
Available online xxxx

Keywords:
Ta films
DOMS
HiPIMS
Film growth
Phase tailoring

ABSTRACT

The effect of energetic ion bombardment on the properties of tantalum thin films was investigated. To achieve such energetic ion bombardment during the process the Ta thin films were deposited by deep oscillation magnetron sputtering (DOMS), an ionized physical vapor deposition technique related to high power impulse magnetron sputtering. The peak power was between 49 and 130 kW and the substrate was silicon at room temperature and ground potential. The directionality and the energy of the depositing species was controlled by changing the ionization fraction of the Ta species arriving at the substrate at different peak powers. In this work, the surface morphology (AFM), microstructure (SEM), structure (XRD) and hardness and Young's modulus (nanoindentation) of the films were characterized. The ion energy distributions (IEDs) were measured using an electrostatic quadrupole ion energy and mass spectrometer (HIDEN EQP 300). The IEDs showed that the DOMS process applies a very energetic (up to 120 eV) ion bombardment on the growing tantalum films. Therefore, with such conditions it was possible to deposit pure α-Ta (of 2 μm of thickness) without the use of additional equipment, i.e., without substrate bias or substrate heating. Conditions are therefore significantly different than in previous works, offering a much simpler and cheaper solution to up-scale for industrial operation.

1. Introduction

Tantalum is a refractory metal with a number of unique characteristics and attractive properties such as low electrical resistivity, high melting point, and excellent chemical inertness at temperatures below 150 °C [1,2]. Due to these characteristics Ta has many applications, for instance as heat and wear resistant protective coatings, as-diffusion barriers in integrated circuits and magnetic disk drives [3–5]. Tantalum exists in two distinct phases: a stable α-phase with a body-centred cubic lattice structure and a metastable β-phase with a tetragonal lattice structure. The tough and ductile α-phase is required in most industrial applications, such as diffusion barrier layers, metallic corrosion protective layers, and in biomedical devices. Bulk Ta metal has the α-phase structure while the β-phase appears in thin films. The β-phase is hard and brittle, and its presence may compromise the film performance. According to the state of the art, formation of the β-phase in magnetron sputtered films can be prevented by manipulating the deposition parameters, such as sputtering gas, energetic ion bombardment, substrate temperature and substrate material [6–10]. Among these, it was found that the use of energetic ion bombardment on the growing film played an important role in the tantalum film structure and properties [11].

In direct current magnetron sputtering (DCMS) the most influential deposition parameter with respect to ion bombardment on the growing film is the substrate bias. However, applying a negative bias to the substrate only allows us to extract process gases (Ar) ions from the plasma as the ionization degree of the sputtered material is very low (e.g., 1%–3%). In recent years, new magnetron sputtering deposition techniques that allow producing highly ionized fluxes of sputtered material have been developed. High peak power is applied to the target for a short period of time causing much higher plasma densities than in DCMS by ionization of the sputtered species by electron impact. Two of these recent developments, called High-power Impulse Magnetron Sputtering (HiPIMS) [12–15], and Modulated Pulsed Power Magnetron Sputtering (MPPMS) [16–18], were already used for the deposition of tantalum thin films. Alami et al. [11] found that HiPIMS allowed us to control the Ta phase formation and established a bias voltage window for deposition of α-tantalum on Si. Lin et al. [19] found that all α-tantalum films are deposited by MPPMS when the negative bias voltage was – 50 V or greater. In both cases the energetic bombardment of the growing film was achieved by substrate biasing. Recently, a new HIPIMS process called deep oscillation magnetron sputtering (DOMS) [20–24] was developed. This process uses large voltage oscillation packets in long modulated pulses for achieving high peak target currents and voltages.

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http://dx.doi.org/10.1016/j.surfcoat.2016.08.017
0257-8972/© 2016 Published by Elsevier B.V.

The authors have shown previously that the DOMS process allowed us to tailor the microstructure and properties of metal and nitride thin films without the need of substrate biasing [20,21,24]. Therefore, it is expected that the energetic ion bombardment in DOMS could also be used to tailor the phase composition and improve the structure and properties of tantalum thin films without the use of substrate bias and/or substrate heating.

In the current study, tantalum thin films were deposited on silicon by DOMS at different peak powers in order to have different levels of ion bombardment. The structure, morphology and mechanical properties of tantalum thin films were characterized. The ion energy distributions (IEDs) were measured using an electrostatic quadrupole plasma mass spectrometer (HIDEN EQP 300) both for DOMS or DCMS process. Five films were deposited by DCMS with the increase of the substrate bias for comparison purposes.

2. Experimental procedure

2.1. Film deposition

Tantalum thin films (of 2 μm of thickness) were deposited on Si (100) substrates using a continuous D.C. power source (Huttinger PFG 7500 DC) and a DOMS power supply (HiPIMS Cyprium™ plasma generator, Zpulser Inc.) at room temperature and ground potential. An example of the DOMS discharge voltage and current wave forms used in this work is shown in Fig. 1. The voltage on-time ($t_{on} = 6$ μs), oscillation period ($T = 50$ μs) and pulse duration ($D = 1250$ μs) were kept constant for all depositions, while the pulse frequency (F) was automatically adjusted by the DOMS power supply software in order to maintain a specified time-averaged power.

The 2 cm × 2 cm substrates were cut from Si (100) wafers. Prior to the depositions they were ultrasonically cleaned in a sequence of acetone and ethanol solutions baths, for 10 min each. They were then glued with silver glue (99.9% purity) onto an aluminium substrate holder and placed in the deposition chamber made from high grade stainless steel with 40 cm × 40 cm × 40 cm dimensions. In all depositions, the substrate-to-target distance and substrate rotation was kept at 80 mm and 23.5 rpm, respectively. The target used consisted of 99.95% pure Ta with an area of 150 mm × 15 mm and 7 mm thickness. A base pressure lower than 4×10^{-4} Pa was achieved before all depositions using a turbomolecular pump. A constant Ar (99.999%) flow rate of 15 sccm was used in all the depositions resulting in a discharge pressure of 0.7 Pa. A constant average target power of 1.2 kW was used for all depositions (DOMS and DCMS) in order to minimize variations of thermal effects during the deposition of the films. The deposition time was changed in order to achieve 2 μm of thickness.

The tantalum thin films were deposited by DOMS with the increase of the peak power (P_p) in order to increase the ionization of the sputtered material during the process. The P_p was varied between 49 and 130 kW by changing the charging voltage (DC_{int}) between 270 and 400 V. The peak power (P_p) is defined as the product $V_p \times I_p$. The main DOMS deposition parameters are compiled in Table 1. Five films were deposited by DCMS with increased negative substrate bias (−30 V, −50 V, −80 V and −120 V) for comparison purposes. In these depositions the voltage target and current target were 333 V and 3.75 A, respectively.

2.2. Ion energy distribution measurements

The ion energy distributions (IEDs) were measured using an electrostatic quadrupole plasma mass spectrometer (HIDEN EQP 300). This equipment can measure up to 1 keV/charge and is assisted by 70 l/s turbo pump for differential pumping of the instrument. The tantalum target with a diameter of 7.6 cm was mounted in front of the EQP orifice (100 μm diameter) with a 10 cm distance between them. An average target power of 600 W was used for both DOMS and DCMS. The IEDs were measured from 0 to 100 V scan voltage with a step size of 0.5 V and a 1000 ms dwell time. The extractor voltage used in EQP measurement was equal to 40 V. In accordance with the reference [25], after the measurements the raw data were corrected for the double charge ions by multiplying the scan voltage by two and dividing the count rate by two to account for the energy bin width. The following ions were analysed: Ta^+, Ta^{2+}, Ar^+, Ar^{2+}. It has to be mentioned that the here-presented distributions merely provide a semi-quantitative description of the energy distributions involved during the DOMS tantalum sputtering process because difficult-to-assess instrument functions such as the acceptance angle distort the measured functions.

2.3. Film characterization

The crystal structure of tantalum thin films was analysed by X-ray diffraction (XRD) (PANalytical X'Pert PRO MPD) using Cu Kα radiation (45 kV and 40 mA) with a parallel beam in θ–2θ geometry. The incident beam optics consisted of a hybrid monochromator (with a Cu W/Si mirror and a double crystal Ge (220)). A parallel plate collimator (0.7°) and Soller slits (0.004°) were mounted on the path of the diffracted beam. A

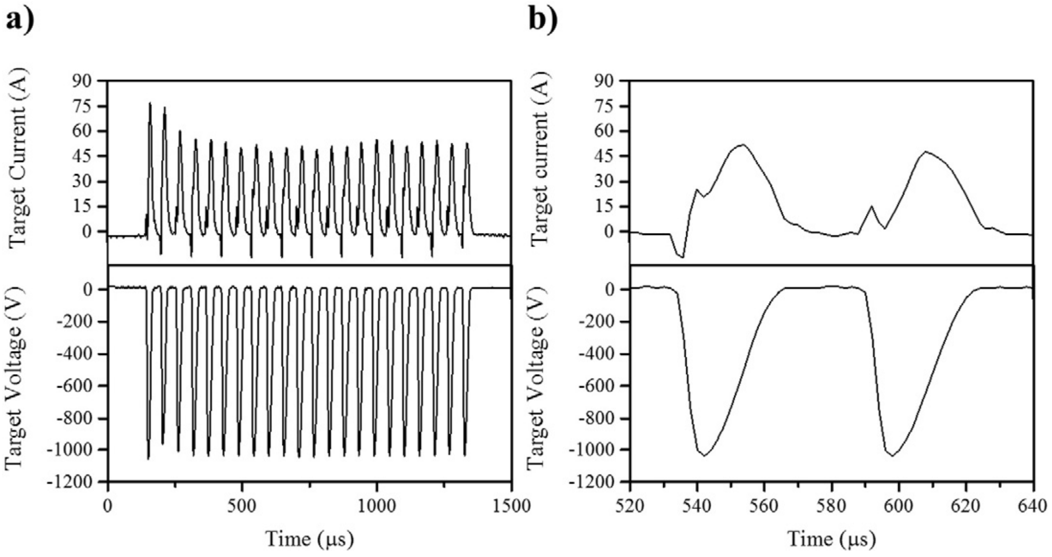

Fig. 1. a) The target voltage and current oscillation waveforms measured during the Ta thin film depositions. b) Small oscillation pulses within one long pulse.

Please cite this article as: F. Ferreira, et al., Surf. Coat. Technol. (2016), http://dx.doi.org/10.1016/j.surfcoat.2016.08.017