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journal homepage: www.elsevier.com/locate/surfcoatInert gas effects on the deposition rate of TiO₂ during reactive HiPIMS

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ABSTRACT

Deposition rates have been measured during the reactive HiPIMS of Ti in the presence of oxygen and different inert gases (i.e. mixtures of X/O₂ where X = Ne, Ar, Kr or Xe) by means of a quartz crystal microbalance (QCM). The QCM was positioned above the erosion racetrack directly facing the target surface at two different axial distances (50 and 100 mm). The HiPIMS discharge was operated with a pulse on-time $\tau = 100 \mu\text{s}$, a pulse frequency $f = 100 \text{ Hz}$ and a constant average discharge power $P_{\text{avg}} = 100 \text{ W}$ (50 W for Xe/O₂). The oxygen partial pressure, p_{O_2} , was maintained at a constant $0.2p_t$ where p_t is the total pressure and was maintained at a constant 0.4 Pa. Using these conditions, the discharge was operated in the so-called 'poisoned' mode. In contrast to the trends predicted by SRIM as well as those measured in DCMS, the power-normalized static deposition rates in reactive HiPIMS of titanium measured in gas mixtures of oxygen were observed to increase with the mass of the inert gas. The observed trend was attributed to a decreased *return effect* as a result of an increased average absolute target potential during the pulse on-phase when employing heavier inert gases as the buffer gas. For the case of Kr/O₂, the normalized deposition rate measured in HiPIMS was found to be 87% of that measured in equivalent DCMS operation.

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1. Introduction

The main drawback of a newly emerging physical vapour deposition technology, high power impulse magnetron sputtering (HiPIMS) [1], is the almost universally reported reduced *power normalized* deposition rate when compared with conventional direct current magnetron sputtering (DCMS). The normalized deposition rate for non-reactive HiPIMS has been observed to be typically 30–85% [2,3] of the value measured during equivalent DCMS operation. There are several mechanisms thought to be responsible for this apparent reduction in deposition rate and they have been the subject of some recent publications [4,5]. Due to the increased ionization of sputtered species that occurs in a HiPIMS discharge there exists a so-called *return effect* whereby a non-negligible fraction of the ionized sputtered material is back-attracted to the target surface [4–6]. Another major issue is the less than proportional scaling of the sputter yield with incident ion energy, often referred to as the *yield effect* [4,7]. HiPIMS typically employs higher absolute target potentials when compared with DCMS and hence lower average ion currents are necessary for the same time-averaged power. The deposition rate is typically proportional to the sputter yield; however the sub-proportional scaling of sputter yield with incident ion energy results in a decreased normalized deposition rate for increased absolute target potential values. Other effects include the ion species effect [4], transport effects [8,9], coating effects [3,4], power-switching effects [4] and magnetic field effects [5]. Anders [4] has presented a detailed treatment of the main mechanisms affecting the deposition rate during non-reactive

HiPIMS. Also, Vlcek and Burcalova developed expressions for deposition rates in a phenomenological model for HiPIMS [10].

In reactive HiPIMS, the composition of the discharge and the extent of the target coverage/poisoning are important aspects to consider. In the case of sputtering oxides, the oxygen reacts with the target surface to varying degrees mostly dependent upon the ion contribution to the discharge current and the reactive gas flow rate resulting in a metallic target partially or completely covered ('poisoned') with a compound layer. It is known that many oxide compounds suffer from a lower deposition rate than their pure metal counterparts [11] and so the reduced normalized deposition rate in HiPIMS when compared with DCMS can be of great concern in reactive sputtering.

Furthermore, large amounts of high energy negative ions are generated during reactive DCMS [12–16] and reactive HiPIMS [17] in the presence of electronegative gases such as oxygen. It is possible that the bombardment of the substrate by the high-energy population of the negative ions will lead to resputtering of the growing film and result in a reduction of the effective deposition rate. Despite this, there are some promising aspects of reactive HiPIMS including hysteresis-free operation [18] and even deposition rates measured to be higher than those achieved in the equivalent DCMS process [18–20]. For example, Sarakinos et al. [18] found that the deposition rate of TiO_x thin films increased by up to 40% for HiPIMS of a TiO_{1.8} target when compared with DCMS. Also, the measured deposition rates during the assisted HiPIMS of titanium have been correlated to the crystal formation phase of the deposited TiO₂ [21], meaning the deposition rate could be potentially used as a control parameter.

The work presented in this contribution focuses on the measured static normalized deposition rates of titanium oxide during reactive

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HiPIMS of titanium in a gas mixture of X/O₂, where X = Ne, Ar, Kr or Xe. The primary mechanisms affecting the deposition rate are briefly discussed and deposition rates measured in reactive HiPIMS are compared to measurements made in nominally equivalent reactive DCMS discharges for $d = 100$ mm.

2. Experimental set-up

2.1. Arrangement of HiPIMS set-up

A cylindrical vacuum vessel with a length of 300 mm and a diameter of 260 mm equipped with a commercial circular planar unbalanced magnetron source (GENCOA Ltd.) was pumped down to a base pressure of approximately 5×10^{-4} Pa using a turbomolecular pump backed by a rotary pump. The magnetron was equipped with a new titanium target (99.95% purity) with a diameter of 75 mm and powered by a HiPIMS power supply built in-house, described in detail in [22].

In addition to the HiPIMS power supply, a low power (≤ 10 W) pre-ionization unit was used to facilitate operation at low pressure and to allow for greater reproducibility of pulse waveforms, which is also described in [22]. The switching of the HiPIMS power supply was triggered by an external pulse generator (Thandar TG105) by which the voltage pulse width τ and frequency f were controlled. In this investigation, the voltage pulse on-time and frequency were maintained constant at $\tau = 100$ μ s and $f = 100$ Hz respectively.

Process gases were introduced into the chamber at flow rates controlled by two independent mass flow controllers (MKS 1179A). The total working gas pressure, $p_t = p_x + p_{O_2} = 0.4$ Pa (X = Ne, Ar, Kr, Xe), was monitored using a capacitance pressure gauge (MKS 628A). All working gases used in this investigation were of a purity of at least 99.995%. A schematic of the experimental apparatus is given in Fig. 1.

To obtain the desired oxygen partial pressure, the oxygen flow rate was increased beyond the hysteresis transition from metallic to poisoned mode and then reduced as necessary to maintain a constant oxygen partial pressure; $p_{O_2} = 0.2p_t$. The target voltage $V_d(t)$ was measured using a high voltage probe (Tektronix P5100) and the discharge current $I_d(t)$ was measured using a DC coupled current probe (Tektronix TCP202) which were both connected to a digital oscilloscope (Tektronix TDS3014, 100 MHz bandwidth). The average discharge power, P_{avg} , was monitored in real-time using the same oscilloscope, calculated as the mean of the product of $V_d(t)$ and $I_d(t)$ and controlled by manually varying the initial target potential V_0 provided by the power supply.

2.2. Deposition rate measurements

Deposition rates, were measured by means of a quartz crystal microbalance (QCM) with the exposed face of the gold-coated sensor crystal (6 MHz) grounded and the opposite side connected to a thin film deposition monitor (Maxtek TM-400, Inficon). The sensor crystal was placed directly above the racetrack facing the target surface at two different axial distances, $d = 50$ and 100 mm. Reading directly from the DAC output of the deposition rate monitor, the film thickness was monitored as a function of time using a voltage probe (Tektronix P6139A) over a period of 500 s. The output range of the monitor was 0 to 5 V, corresponding to a thickness range of 0 to 100 Å (i.e. a step of 0.05 V/Å). For calculation of the film thickness, the deposited film was assumed to be stoichiometric titania with a density of 4.26 g/cm³. Prior to any deposition rate measurements, the discharge was operated in metallic mode with a pressure of 0.93 Pa in the presence of the relevant inert gas (zero oxygen flow) for 15 min in order to sputter-clean the target surface. Following the introduction of oxygen to the desired partial pressure, the discharge was operated for a further 15 min to obtain an equilibrium state. During this run-in procedure, the QCM was protected by a stainless steel shield, which could be removed without breaking vacuum. The same initial procedure was employed for each deposition rate measurement.

3. Results and discussion

3.1. HiPIMS pulse waveforms

Waveforms of the target discharge current $I_d(t)$ and potential $V_d(t)$ for the different gas mixtures (X/O₂ where X = Ne, Ar, Kr or Xe) are shown in Fig. 2. The discharge parameters were as follows: $P_{avg} = 100$ W, $f = 100$ Hz, $\tau = 100$ μ s, $p_t = 0.4$ Pa and $p_{O_2} = 0.2p_t$. For the case of Xe/O₂, the average discharge power was maintained at 50 W due to the power supply being unable to maintain an average discharge power of 100 W without lengthening the pulse on-time τ or increasing the pulse frequency f . For an increased mass of the sputtering ion, the Bohm velocity naturally decreases thereby increasing the transit time across the cathode sheath. In addition, and as discussed below, the reduction in the secondary electron emission coefficient for heavier inert gases explains the observed increase in the time taken for $I_d(t)$ to reach a maximum for increasing inert gas mass mixed with oxygen.

Also evident from Fig. 2 is a clear increase in required absolute target potential for heavier gas mixtures to maintain a constant discharge

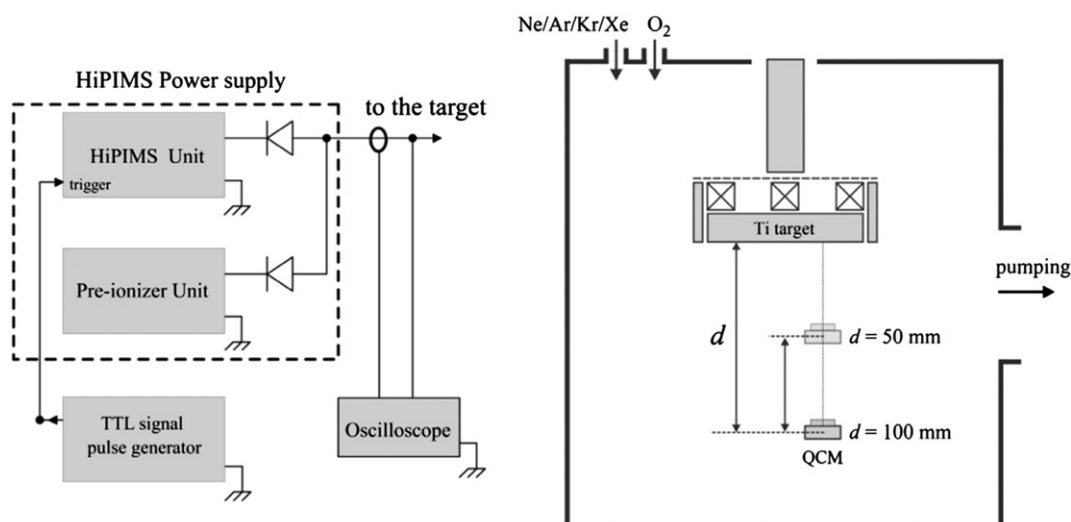


Fig. 1. Schematic representation of the experimental set-up. The QCM was moved axially with respect to the target into two positions; $d = 50$ to 100 mm.

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