Influence of sputtered species ionisation on the hysteresis behaviour of reactive HiPIMS with oxygen admixture

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Abstract

In this paper, the hysteresis behaviour of a reactive magnetron sputtering process with oxygen admixture is studied by both experiment and model. The ground state number densities of titanium atoms and ions, and the deposition rates in high power impulse magnetron sputtering discharge were determined for a constant average applied power and pulse duration, while the repetition frequency and reactive gas supply were varied. The hysteresis curve reduced in width and shifted towards the lower oxygen supply with a decrease in the repetition frequency. These experimentally observed trends were well reproduced by a modified Berg model. The presented model utilised measured ionisation fraction of sputtered species and considered the back-attraction of the ionised sputtered species to the target. Significance of the back-attraction process was observed as large fraction of ionised sputtered particles was found to be lost back to the target. This drastically decreased the number of particles deposited onto the substrate, which reduced not only the deposition rate, but also the gettering of the reactive gas. Consequently, the simulated hysteresis curve was shifted and reduced in width as it was observed experimentally.

Keywords: magnetron sputtering, reactive HiPIMS, ionisation fraction, titanium, oxygen, model

1. Introduction

Sputtering of metal targets in a reactive gas atmosphere results in the deposition of compound coatings including hard and protective thin films such as TiN [1, 2] and TiAlN [3], optical coatings such as TiO₂ [4, 5] and ZrO₂ [6, 7] or transparent conductive oxides coatings such as Al-doped ZnO [8, 9]. The deposition of compound films is a complex process which might exhibit a hysteresis behaviour [10]. Hysteresis in reactive direct current magnetron sputtering (dcMS) discharge has been studied extensively and it is well described [10–14]. In contrast, reactive high power impulse magnetron sputtering (HiPIMS), which utilises high ionisation of sputtered species [15–18] to synthesise coatings with improved quality [1, 19, 20] has still not been fully described and understood.

The hysteresis behaviour of reactive HiPIMS with oxygen and nitrogen admixtures was investigated by Čapek et al [21] and Kadlec et al [22]. Both experiment and model demonstrated that critical pumping speed in HiPIMS is lower compared to dcMS, which implies a reduction in the hysteresis curve. Furthermore, HiPIMS discharge with oxygen admixture has been thoroughly modelled by several means by different groups [15, 17, 23]. Experimentally, the evolution of plasma density and effective electron temperature with an oxygen mass flow in reactive HiPIMS discharge was determined by Čada et al [24]. Using the Langmuir probe, it has been observed that effective electron temperature increases with oxygen supply and that plasma density evolution correlates with the waveform of the discharge current. Additionally, the time-resolved number densities of the sputtered species, argon and oxygen were measured in a reactive HiPIMS discharge by Britun et al utilising optical emission, absorption and laser-based spectroscopic techniques [16, 25].
The aim of this paper is to study the impact of the high ionisation fraction of sputtered species on the hysteresis behaviour of reactive HiPIMS discharge. The ionisation fraction of sputtered species was varied with the change in repetition frequency. An optical emission spectroscopy was utilised to determine the number densities of sputtered particles and consequently to derive the ionisation fraction of sputtered species [26]. The deposition rates were evaluated from thicknesses of deposited coatings. The reactive HiPIMS discharge was also modelled by modified Berg model to assess the effect of the ion back-attraction on the hysteresis curve’s shape.

2. Experimental setup

The experimental setup is depicted in figure 1. The experiments were performed using an Alcatel SCM 650 industrial magnetron sputtering system. Its deposition chamber has a cylindrical shape with a diameter of 650 mm and height of 350 mm. An unbalanced magnetron source (see figure 2) manufactured by HVM Plasma was mounted at the bottom of the chamber. A rectangular titanium target (99.7% purity) with dimensions of 250 × 73 mm was sputtered during the experiments, which resulted in a racetrack area of
approximately 8800 mm$^2$ and racetrack length of approximately 300 mm.

The deposition chamber was pumped by a roots and a turbo-molecular pumps to the base pressure below $10^{-4}$ Pa. The residual pressure was determined by a compact full vacuum range gauge PKR 251 (Pfeiffer LTD). Argon gas (purity of 99.996%) was fixed at a constant mass flow of 125 sccm. It resulted in 5 Pa working pressure as measured by the MKS Instruments 390HA Baratron with the standard deviation of 0.08% of reading as stated by the manufacturer. Oxygen (purity of 99.995%) mass flow was regulated by a Genco LTD Speedfio Mini fast feedback system [27, 28]. The utilisation of the Speedfio Mini allowed for controlled operation within the hysteresis curve transition region of the reactive magnetron sputtering. The cathode voltage served as an input signal for the fast feedback system.

Melec SIPP 2000 HiPIMS generator powered the discharge either in the dcMS or in the HiPIMS operation mode. The averaged applied power was set to 1 kW at all times. The HiPIMS experiments were performed at repetition frequencies of 80, 100, 120, 150 and 200 Hz, the pulse duration was always kept at 200 $\mu$s. The power generator was outfitted with a voltage probe and a current probe for time-resolved electrical measurements. The cathode voltage and the discharge current waveforms were recorded by the Tektronix TDS 2014C digital oscilloscope. The process parameters are summarised in Table 1.

| Parameter                  | Value                              |
|----------------------------|------------------------------------|
| Target                     | Ti (250 × 73 mm)                   |
| Working pressure           | 5 Pa                               |
| Reactive gas               | Oxygen                             |
| Operation mode             | dcMS or HiPIMS                     |
| Average applied power      | 1 kW                               |
| HiPIMS pulse duration      | 200 $\mu$s                         |
| HiPIMS repetition frequency | 80, 100, 120, 150, 200 Hz           |
| Studied region             | 8 mm above target                  |
| Deposition time            | 120 or 180 min                     |

Table 1. The main process parameters.

The hysteresis curve contains two critical points, which separate metallic, transition and poisoned modes [10]. The oxygen gas reacts with all metallic surfaces, where it forms a compound layer. The compound coverage enlarges with increase in the partial pressure of reactive gas. Compound coverage of the target surface becomes significant at the first critical point, which results in the lower gettering capacity of the system due to the lower amount of metal particles sputtered from the target. Thus, a lower supply of reactive gas is needed in the transition mode to further increase the compound coverage of the target. From the second critical point, the compound almost completely covers all the surfaces in the deposition chamber.

The widest hysteresis curve is observed in reactive dcMS. In the case of reactive HiPIMS, the hysteresis curve shifts towards the lower oxygen supply and the hysteresis...
The cathode voltage is higher in the poisoned mode compared to the metallic mode due to the lower secondary electron emission yield of titanium-oxygen compound compared to the secondary electron emission yield of the pure titanium surface [33]. The cathode voltage difference between the metallic and the poisoned mode decreases with a decrease in the repetition frequency.

The temporal evolution of the cathode voltage has a rectangular shape with a rise time equal to the fall time of 600 ns. Typical time evolutions of the discharge current are shown in figure 4. The depicted discharge current waveforms were obtained during the HiPIMS discharge with the repetition frequency of 80 Hz and different degrees of target poisoning as represented by the different cathode voltages. The waveform obtained in the metallic mode of reactive HiPIMS (474 V) has a fast initial discharge current rise, followed by a tendency for saturation. The typical waveform detected in the transition mode (510 V) has an approximately triangular shape with a constant slope. In the poisoned mode (536 V), an initial slow increase in discharge current is followed by a steep rise. The discharge current waveforms in reactive HiPIMS were thoroughly studied by Gudmundsson et al [15] and Brenning et al [34]. They found that the evolution of the discharge current depends on two recycling mechanisms. In the metallic mode the recycling of sputtered particles is promoted, leading to the saturation of the discharge current. However, in the poisoned mode the working gas recycling is dominant, which leads to a steep current increase in the later part of the HiPIMS pulse.

The peak discharge current attained at the end of the HiPIMS pulse increases with an increase in the degree of target poisoning. However, the mean discharge current determined from the integral of the discharge current waveform decreases. As a result of decreasing the repetition frequency of the HiPIMS discharge, the peak discharge current increases and the mean discharge current decreases for the same degree of target poisoning.

The ground state number densities of titanium atoms [Ti] in both dcMS and HiPIMS and titanium ion [Ti⁺] in HiPIMS discharges for different oxygen gas flows are shown in figures 5(a) and (b), respectively. In the case of dcMS, in which [Ti⁺] was under the detection limit of the EBF method, the ionisation fraction of sputtered species is barely a few percent [35, 36]. Titanium number densities decrease with the increase in degree of target poisoning due to the lower sputtering yield of the compound compared to the sputtering yield of titanium [10]. In the metallic mode of dcMS at oxygen supplies up to 5.3 sccm a peculiar increase of [Ti] was measured. It might be caused by the quenching of the titanium metastable states by reactive gas, as the number density at these states can reach tens of percent of the ground state number density [37, 38].

The lower the repetition frequency of HiPIMS discharge, the lower [Ti] and the higher [Ti⁺] is detected for the same degree of target poisoning. Generally at constant average power, lower repetition frequency implies higher average...
power delivered per one pulse and thus denser plasma and a higher probability of sputtered species ionisation.

Selected evolutions of sputtered species ionisation fractions are depicted in figure 6. The sputtered species ionisation fraction for 200 Hz repetition frequency increases with the increase in the degree of target poisoning. The increase in the ionisation fraction of sputtered species is attributed to the combination of three effects as discussed in our previous paper [18]—the longer residual time of sputtered species in the magnetised plasma, the higher electron temperature and the higher electron density in the poisoned mode compared to the metallic mode. The ionisation fraction of sputtered species is approximately constant in the case of reactive HiPIMS discharge with the repetition frequency of 80 Hz at which the maximal peak discharge current is attained.

The discharge parameters of reactive HiPIMS with 80, 200 Hz and reactive dcMS in metallic, transition and poisoned mode are summarised in table 2 and were discussed above. The condition without any oxygen is taken as the metallic mode, the transition mode is represented by the condition measured approximately in the middle of the transition region, and the condition after the second critical point stands for the poisoned mode.

4. Modelling of reactive dcMS and HiPIMS discharge

The effect of the sputtered species ionisation on the hysteresis behaviour of both the dcMS and HiPIMS discharge with reactive gas admixture is simulated using a simple time-averaged model. The time-averaged model may describe even HiPIMS discharge where the surface processes are dominant and thus require much longer time than a single typical HiPIMS pulse provides [39]. The proposed model for HiPIMS is based on the Berg model [11–13] commonly used for the modelling of hysteresis behaviour in reactive dcMS. The Berg model compiles balance equations at the target and substrate level in steady-state conditions. The equation for the target poisoning calculates the balance between the formation and out-sputtering of the compound part of the target. At the substrate level, the equilibrium between the formation of metallic and compound part of the substrate is calculated. The supplied reactive gas forms the compound at the target, substrate and chamber walls through the process of chemisorption and also induces an increase in the partial pressure of reactive gas depending on the capacity of the pumping system. The used Berg model does not account for the implantation of reactive gas ions into the target and the reactive gas knock-in into the target as discussed in [13].

The presented model enhances the Berg model by taking into account the back-attraction of ionised sputtered metallic species to the target together with the sputtering induced by these ionised sputtered particles. It is assumed that the back-attracted ionised metallic species either sputter or do not change the conditions at the target surface. The sputtered metallic species, which are not back-attracted to the target, are transported to the substrate. The sputtered reactive gas particles are assumed to return to the gas phase. The diagram of the proposed model for reactive HiPIMS discharge is shown in figure 7.
A magnetron cathode is bombarded by both argon and titanium ions resulting in the sputtering of the target material. The number of sputtered particles depends on the number, energy and mass of the impinging ions. The total number of metal atoms sputtered from the target surface per second is

\[ F_m = \frac{J_g}{q} [Y_{mmg}(1 - \Theta_t) + Y_{mcg} \Theta_t] A_t \]

\[ + \frac{J_m}{q} [Y_{mmm}(1 - \Theta_t) + Y_{mcg} \Theta_t] A_t, \]

where \( J_g \) and \( J_m \) are the current densities induced by working gas and metal ions, respectively, \( q \) is the elementary charge. \( Y_{mmg} \) and \( Y_{mcg} \) are the sputtering yields of metal from metallic and compound part of target by working gas ions, respectively and \( Y_{mmm} \) and \( Y_{mcg} \) are the sputtering yields of metal from metallic and compound part of target by metal ions, respectively. \( \Theta_t \) stands for a poisoned fraction of the target and \( A_t \) is the target surface area. The first term on the right-hand side of the equation (1) describes sputtering induced by working gas ions, and the second term refers to the sputtering caused by back-attracted metal ions.

The total number of sputtered metal ions back-attracted to the target per one second is

\[ F_b = \frac{J_m}{q} \alpha \beta A_t \]

where \( \alpha \) is the ionisation probability of the sputtered species and \( \beta \) is the probability of back-attraction to the target.

The total number of reactive gas atoms sputtered from the target surface per second is

\[ F_c = \frac{J_g}{q} Y_{rcg} \Theta_t A_t + \frac{J_m}{q} Y_{rcm} \Theta_t A_t \]

\[ = \frac{J_g}{q} Y_{rcg} \Theta_t A_t + F_b Y_{rcm} \Theta_t, \]

where \( Y_{rcg} \) and \( Y_{rcm} \) are the sputtering yields of the reactive gas atoms from compound part of target by working gas and metal ions, respectively.

Current densities \( J_g \) and \( J_m \) can be derived from the discharge current \( I_D \). The discharge current is a sum of the ion current of the working gas and of the metal, while the current of secondary-emitted electrons is neglected. To obtain the

| Mode          | Rep. Freq. (Hz) | O₂ Supply (sccm) | O₂ part. Pressure (Pa) | Cath. Voltage (V) | Mean Curr. (A) | Max. Curr. (A) | [Ti] (×10¹⁷) | [Ti⁺] (×10¹⁷) | IFSS % |
|---------------|----------------|------------------|------------------------|-------------------|----------------|----------------|----------------|----------------|--------|
| Metallic      | 80 Hz          | 0.0              | 0.000                  | 474               | 2.1            | 204            | 0.74 ± 0.02   | 3.44 ± 0.10   | 82     |
|               | 200 Hz         | 0.0              | 0.000                  | 378               | 2.7            | 110            | 0.91 ± 0.02   | 1.63 ± 0.04   | 64     |
|               | dcMS           | 0.0              | 0.000                  | 278               | 3.6            | —              | 1.32 ± 0.06   | —              | —      |
| Transition    | 80 Hz          | 1.8              | 0.059                  | 510               | 2.0            | 340            | 0.12 ± 0.01   | 0.60 ± 0.02   | 83     |
|               | 200 Hz         | 2.9              | 0.061                  | 466               | 2.1            | 126            | 0.22 ± 0.01   | 0.78 ± 0.03   | 78     |
|               | dcMS           | 4.4              | 0.075                  | 397               | 2.5            | —              | 0.35 ± 0.02   | —              | —      |
| Poisoned      | 80 Hz          | 3.4              | 0.135                  | 536               | 1.8            | 416            | 0.02 ± 0.01   | 0.13 ± 0.01   | 84     |
|               | 200 Hz         | 3.4              | 0.125                  | 508               | 2.0            | 170            | 0.02 ± 0.01   | 0.14 ± 0.01   | 86     |
|               | dcMS           | 4.9              | 0.211                  | 432               | 2.3            | —              | 0.04 ± 0.01   | —              | —      |

Figure 7. Diagram of the model for reactive HiPIMS.
metal ion current, several sputtering cycles are computed. The calculation starts for only argon ion bombardment of the target which sputter a certain amount of metal atoms. Part of these metals is ionised, back-attracted to the target, where they contribute into sputtering. When an equilibrium between the argon, metal bombardment and metal out-sputtering is reached, the metal ion current is obtained.

The target steady-state condition represents the balance between the target poisoning and out-sputtering of reactive gas atoms:

$$\epsilon \eta \Gamma (1 - \Theta_s) A_s = \frac{\sigma}{\epsilon} F_c,$$  

(4)

where $\epsilon$ stands for the number of compound molecules formed from a reactive gas molecule, $\eta$ is a sticking coefficient, $\Gamma$ is a flux of reactive gas molecules towards a surface and $\sigma$ stands for a number of reactive gas atoms in a reactive gas molecule.

The chemisorption of reactive gas molecules and the deposition of metallic particles is determined at the substrate level. The steady-state equation at the substrate level is defined as

$$\epsilon \eta \Gamma (1 - \Theta_s) A_s = (F_m - F_i) \Theta_s,$$  

(5)

where $\Theta_s$ denotes the poisoned fraction of the substrate and $A_s$ is the substrate surface area. The left-hand side of equation (5) represents compound formation at a metallic fraction of the substrate surface, and the term at the right-hand side stands for the deposition of metallic particles at the compound fraction of the substrate. Metals deposited on the metallic fraction of the substrate do not change the conditions at the substrate surface and therefore are neglected.

Measured evolutions of the reactive gas partial pressure as a function of the reactive gas supply are simulated for the dcMS and the HiPIMS. All the input parameters are either measured or estimated based on the values reported in other scientific papers. The following parameters were measured and used in the model: the target surface area $A_t = 182.5 \text{ cm}^2$, substrate surface area $A_s = 5000 \text{ cm}^2$ and pumping speed $S = 0.06 \text{ m}^3 \text{s}^{-1}$. The sputtered species ionisation probability $\alpha$ is assumed to be the same as the measured ionisation fraction of sputtered species shown in figure 6. The temperature of 500 K and a sticking coefficient $\eta = 1$ are assumed constant for all modelled cases. The coefficient $\sigma$ equals 2 as oxygen molecule contains two oxygen atoms. The formation of a TiO$_2$ compound is assumed, and thus, the coefficient $\epsilon$ equals 1.

The probability of back-attraction of the sputtered metal ions is estimated to be 0.7 for all the studied HiPIMS conditions. The value of back-attraction probability is close to the value of 0.9 used for the simulation by Gudmundsson et al [15]. The sputtering yields of metal from metallic fraction of the target $Y_{mcg}$ and $Y_{mcm}$ are calculated based on the work of Matsunami et al [40] and the ion energy determined by the measured cathode voltage shown in figure 3(b). The sputtering yields $Y_{mcg}$ and $Y_{mcm}$ are assumed to be 30 times lower compared to $Y_{mng}$ and $Y_{mmn}$, respectively [41]. Preferential sputtering of oxygen from compound part of the target is taken into account [13], the sputtering yields $Y_{rcg}$ and $Y_{rcm}$ are assumed to be 1.5 times higher compared to $Y_{mcg}$ and $Y_{mcm}$, respectively.

Typical evolutions of measured and modelled O$_2$ partial pressure as a function of the oxygen mass flow are shown in figure 8 for both dcMS and HiPIMS. The shift of critical points and the reduction of the hysteresis curve width in HiPIMS compared to dcMS are clearly simulated. The comparison of the measured and modelled critical points is depicted in figure 9 for all the repetition frequencies studied. Decreasing the repetition frequency, the critical points shift more towards the lower oxygen supply. The shift of the first critical point with repetition frequency is more pronounced compared to the shift of the second critical point as was also reported by Strijckmans et al [23]. Modelled critical points fit well with the measured ones in both dcMS and HiPIMS case.

The effect of the ionised sputtered species’ back-attraction to the target results in lower metal flux to the substrate.
Thus, the getter capacity of the deposition system decreases and the same degree of target poisoning is reached at lower oxygen supplies. It consequently shifts the first and second critical points towards lower oxygen gas flows in HiPIMS compared to dcMS. Lower metal flux to the substrate explains also the lower deposition rate of HiPIMS compared to dcMS [42]. Measured and modelled deposition rates are shown in figure 10 as a function of oxygen supply for both dcMS and HiPIMS. The measured deposition rates are derived from the thickness of deposited films. Modelled deposition rates are calculated from the fluxes of metal and reactive gas particles towards the substrate. The deposition rates are normalised, the deposition rate in dcMS without oxygen admixture is set to be 1 for both experiment and model. The deposition rate slightly increases in the metallic mode due to the incorporation of oxygen into the coating [43]. Afterwards, it decreases with the increase in degree of target poisoning due to the lower sputtering yield of the compound compared to the sputtering yield of titanium [10]. In HiPIMS, the decrease of deposition rate with decreasing repetition frequency is observed for both experiment and model. Modelled normalised deposition rates fit well with the measured ones in dcMS and HiPIMS cases.

The overall agreement between the measured and modelled critical points and normalised deposition rates confirms that the back-attraction of sputtered metal ions is an essential effect for the hysteresis curve width reduction, for the critical points shift and for the reduction of the deposition rate in HiPIMS compared to the dcMS. The findings from the presented model estimate the probability of back-attraction $\beta$ to be constant for all our experimental conditions. Decreasing the repetition frequency and thus increasing the average power per pulse leads to an increase in the titanium ion number density and accordingly in the ionisation fraction of sputtered species. Because the probability of the ion back-attraction was found to be the same for all tested HiPIMS pulses, the same relative portion of the ions is always back-attracted to the target and the rest is transported towards the substrate. Higher ionisation fraction of sputtered species in the magnetised plasma thus implies higher number of sputtered ions attracted back to the target, higher amount of ions transported to the substrate, but a drop in the deposition rate.

5. Conclusions

A systematic study of the hysteresis behaviour of magnetron discharges with various oxygen admixtures operated at the same average applied power is presented. The discharge conditions were gradually changed from dcMS to HiPIMS through the change of repetition frequency. The hysteresis curve shifts to lower oxygen gas supply and reduces in width as the repetition frequency decreases. The ground state number densities of titanium atom [Ti], in both dcMS and HiPIMS and titanium ion [Ti$^+$] in HiPIMS discharges for different oxygen gas supply, are determined from the optical-emission signal utilising the EBF method. As the repetition frequency of HiPIMS discharge decreases, the lower [Ti] and higher [Ti$^+$] is measured for the same degree of target poisoning due to the increase in plasma density and thus increase in ionisation probability.

The hysteresis curves shape is simulated utilising a modified Berg model. The back-attraction of ionised sputtered metallic species to the target is incorporated into the Berg model, ionisation fraction of sputtered species determined from the measured number densities served as one of the input parameters. The back-attraction of the sputtered particles implies lower flux of sputtered particles to the substrate, which causes a decrease in the deposition rate and the getter capacity of the system. It results in a shift and a width reduction of the hysteresis curve. Modelled critical points of hysteresis curves and deposition rates match the measured ones well, proving that the back-attraction of sputtered species to the target is the main effect inducing the critical points shift, hysteresis curve width reduction and reduction of the deposition rate in HiPIMS compared to the dcMS.

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