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Observation of a periodic runaway in the reactive Ar/O\textsubscript{2} high power impulse magnetron sputtering discharge

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This paper reports the observation of a periodic runaway of plasma to a higher density for the reactive discharge of the target material (Ti) with moderate sputter yield. Variable emission of secondary electrons, for the alternating transition of the target from metal mode to oxide mode, is understood to be the main reason for the runaway occurring periodically. Increasing the pulsing frequency can bring the target back to a metal (or suboxide) mode, and eliminate the periodic transition of the target. Therefore, a pulsing frequency interval is defined for the reactive Ar/O\textsubscript{2} discharge in order to sustain the plasma in a runaway-free mode without exceeding the maximum power that the magnetron can tolerate.

I. INTRODUCTION

High power impulse magnetron sputtering (HiPIMS) has been extensively studied and is well known as an energetic ionized physical vapor deposition (IPVD) technique.\textsuperscript{1–5} A high electron density results from the pulsing of the target at high voltage while the duty cycle of the applied voltage is kept low.\textsuperscript{6} This leads to a high ionization level of sputtered materials,\textsuperscript{7} which results in an enhanced density,\textsuperscript{8,9} adhesion improvement,\textsuperscript{10} and surface roughness modification\textsuperscript{11,12} of the grown films.

The HiPIMS plasma runaway to high discharge currents has been thoroughly investigated for non-reactive discharges.\textsuperscript{13–20} Plasma runaway of reactive discharge\textsuperscript{21–24} has been reported in which the ‘runaway’ discharge currents have been initiated with a significant delay compared to the voltage pulse rise time while the voltage pulse was not sometimes discharged properly after the pulse ended,\textsuperscript{22} which can be due the plasma instability caused by usage of very short pulses. Furthermore, the dynamics of the reactive deposition of oxides have been investigated for different materials in a low energy regime of sputtered ions\textsuperscript{25,26} and particularly HiPIMS discharge of a Nb target for various O\textsubscript{2} flows and pulse repetition frequencies in a high energy regime of ion sputtering.\textsuperscript{27,21} It has been proposed that a change in the emission of secondary electrons, as the target transitions from metal mode to oxide mode (depending on the ion energy), is the dominant process influencing the sputtering process.

We have previously reported on the current-voltage-time characteristics of Ar and Ar/O\textsubscript{2} discharges produced by the HiPIMS process.\textsuperscript{28} A pulse repetition frequency range of 15 – 50 Hz was used to study the effect of the pulsing rate on the initiation of the discharge current. Lower pulse repetition frequencies result in a longer off-time between pulses and cause the oxidation to be faster than the etch process of the formed oxide materials on the target. This consequently delays the initiation of the plasma when the next pulse is applied. The same is true when the applied voltage to the target is reduced which results in a delayed rise of the discharge current with respect to the rise edge of the voltage pulse. A transition of the target from metal mode to oxide mode has been understood as...
the reason for the delayed discharge current, which can be related to the emission of the secondary electrons during the pulses.22,28

In this work, we introduce O\textsubscript{2} gas to the Ar discharge step-by-step in order to assess the influence of target poisoning on the current-voltage-time characteristics and the average power of the Ar/O\textsubscript{2} discharge. At a certain O\textsubscript{2} flow, a periodic runaway of the reactive discharge is observed which will be elucidated in terms of variable emission of secondary electrons.

II. EXPERIMENTAL SETUP

A custom-built magnetron sputtering chamber was used to perform all the plasma power and current-voltage measurements. Prior to plasma ignition the base pressure of the chamber was $1 \times 10^{-6}$ Pa. The sputtering gas was argon with purity of 99.999% in Ar discharge, and argon with purity of 99.999% mixed with oxygen gas of 99.999% purity for the reactive Ar/O\textsubscript{2} discharge. The argon flow rate was $q_{\text{Ar}} = 37$ sccm = 100% and the oxygen flow rate was varied between $q_{\text{O}_2} = 0.74$ sccm = 2% and $q_{\text{O}_2} = 1.85$ sccm = 5% for the reactive plasma to study the effect of target poisoning on the plasma discharge. An adjustable throttle valve was used to modify the total gas pressure to 0.7 Pa prior to the ignition of the plasma. For the rest of the experiment the throttle valve was kept unchanged. A Ti target of 99.995% purity and 3 inches (75 mm) in diameter was used in a planar magnetron configuration to produce the plasma. The details of chamber geometry are illustrated in FIG. 1.

The HiPIMS power was supplied by a SPIK1000A pulse unit (Melec GmbH) operating in the unipolar negative mode at constant voltage, which in turn was charged by a dc power supply (ADLGS30). The discharge current and voltage were monitored using a combined current transformer and voltage divider unit (Melec GmbH). LabVIEW software acquired the data and calculated the average power using the measured discharge current and voltage for each applied pulse and the set pulse repetition frequency. The pulse repetition frequency was varied from 140 to 400 Hz for the Ar and Ar/O\textsubscript{2} discharges in order to probe the different oxidation levels on the target surface. The pulse length was 200 $\mu$s for both the non-reactive and reactive plasma. The cathode discharge voltage was set to 700 V in order to be able to sustain the plasma when the target is either at metal mode or oxide mode for various pulse repetition frequencies in the Ar/O\textsubscript{2} discharge.24

III. RESULTS AND DISCUSSION

In order to investigate the influence of target poisoning, an Ar discharge was started at 140 Hz and stabilized after opening the shutter. The initial sudden drop of the power corresponds to the exposure

![FIG. 1. A schematic of the chamber geometry illustrating the positions of the targets and shutters. The grey rectangles show the positions of the 3 inch (75 mm) diameter targets.](image)
of the plasma to a larger volume of the sputtering chamber compared to the confined plasma for a closed shutter. Namely, a confined plasma causes less rarefaction to occur while after shutter opening the sputter wind can push away sputtering gas and result in more rarefaction.\textsuperscript{13} A slight increase of the power after opening the shutter is attributed to the gradual cooling of the target surface which has become hot during the closed shutter due to the confined plasma in front of the target.\textsuperscript{13,29} Subsequently, O\textsubscript{2} gas was applied step-by-step to the discharge, as shown in FIG. 2(a). As the O\textsubscript{2} gas flow increases, the average power begins to decrease due to the target poisoning.\textsuperscript{26,30} At a flow of 5%-O\textsubscript{2}, the average power reduces faster, and eventually the plasma discharge appears to be unsustainable. However, after reaching a very low power level it increases suddenly even above the initial average power, where the target was at metal mode. This process continues as a slow and intense oscillation in the average power which decays out to a stable plasma as the pulse repetition frequency is increased up to 400 Hz (see FIG. 2(b)).

Runaway of plasma to a high density discharge has been previously reported for high sputter yield materials,\textsuperscript{13} transient metals with moderate sputter yields,\textsuperscript{13} and even low sputter yield materials\textsuperscript{20} and oxidized targets.\textsuperscript{21} Furthermore, an unusual increase of discharge current in a reactive discharge from a highly oxidized Ti target has been reported.\textsuperscript{22,23} A generalized criterion for runaway ($\Pi$) to

![Graph showing Ar/O\textsubscript{2} discharge](image)

FIG. 2. (a) Average power of plasma discharge for increasing O\textsubscript{2} flow rate. After applying a 5% flow a drastic drop occurs, which continues to an intense and slow oscillation in the average power. (b) The amplitude of oscillations is affected by the pulse repetition frequency, decreasing as the frequency increases from 140 to 400 Hz. Inset: An enlarged part of the oscillations at a pulse repetition frequency of 200 Hz. $\Delta T$ corresponds to the period of the oscillations and $P_{\text{high}}$ and $P_{\text{low}}$ are related to the maximum and minimum power during the oscillations.
take place can be given by:

$$I = \sum_j (I_{j(i)} + I_{SE(j)}) = \sum_j (1 + \gamma_{SE(j)})I_{j(i)},$$

(2)

where $\alpha_j$ is the effective probability of ionizing atom $j$, $\beta_j$ is the effective probability of returning ion $j$ to the target, and $\gamma_j$ is the sputter yield of ion $j$ when it bombards the target. These generalized fluxes contain both target and gas species, either atoms or ions.\(^{20}\) Anders et al.\(^{20}\) proposed that the gas recycling in front of the target is the dominant mechanism for the runaway to a high current, where the magnetic presheath can accommodate many volts of potential drop and thus defines the ionization zone\(^{31}\) to produce new gas ions. Therefore, a runaway of plasma to a high density discharge is generated mainly from the recycled gas ions for the low sputter yield materials including poisoned targets. It has also been demonstrated that the emission of secondary electrons from the target surface can play an important role for the ionization of re-attracted gas or metal ions.\(^{20}\)

Considering the ionization energies of Ti\(^{+}\) (6.82 eV), Ti\(^{2+}\) (13.58 eV), Ar\(^{+}\) (15.76 eV),\(^{14}\) O\(^{+}\) (13.61 eV),\(^{32}\) and O\(^{2+}\) (12.06 eV)\(^{33}\) the singly and doubly charged metal ions, as well as singly charged reactive gas ions can be present in the plasma as their ionization energy is lower than that of Ar\(^{+}\). The presence of singly and doubly charged Ti ions was reported by Ehiasarian et al.\(^{34}\) for different distances from the target surface. Aiempanakit et al.\(^{35}\) also demonstrated the energy distribution function of O\(^{+}\) for different pulse repetition frequencies. They showed that the energy tail reaches up to 100 eV at lower frequencies where the applied voltages were higher (between 550 – 650 V) in order to keep the average power constant at 100 W. This results in the produced ions being more energetic at higher applied voltages. This directly implies the existence of high-energy O\(^{+}\) ions in the case of this experiment where the applied voltage is higher (700 V) and the frequency is lower (140 – 400 Hz). Ref. 22 also shows the presence of energetic O\(^{+}\) ions when the discharge current reaches a high value for the highly oxidized target. Moreover, for the very similar pulse length (200 $\mu$s) and voltage (700 V), Hála et al.\(^{27}\) have shown the presence of intense emission of O\(^{+}\) and O\(^{2+}\) ions even 100 $\mu$s after the initiation of the pulse close to Cr target surface which indicates the high rate of oxygen dissociation by energetic electrons. Analogously, presence of such high energy ions is expected in this experiment.

Using the concept of the recycled gas ions contribution to the total discharge current, one can write the target current as the sum of gas (argon and oxygen) and target ion currents to the target and the resulting current of secondary electrons from their impacts, proposed by Anders et al.\(^{20}\):

$$I = \sum_j (I_{j(i)} + I_{SE(j)}) = \sum_j (1 + \gamma_{SE(j)})I_{j(i)},$$

(2)

where $\gamma_{SE(j)}$ is the secondary electron emission coefficient resulted from the $j$-type ion impact. Generally, $\gamma_{SE(j)}$ is around 0.1 but it can significantly influence the probability of ionization. However, Phelps and Petrović\(^{36}\) have illustrated that the emission of secondary electrons increases with the increased ion impacting energy. For energies higher than 300 eV, the emission of the secondary electrons from a fully oxidized target surface elevates faster compared to the clean surface.\(^{36}\) This has been attributed to the kinetic electron emission as a dominant process for ejection of the electrons where the high energy of the impacting ions is distributed in lattice vibrations and consequently a thermal equilibrium is established by the ejection of electrons through the thermionic effect.\(^{26,37}\) Therefore, a relatively high emission of secondary electrons from a fully oxidized target (> 0.1) can lead the plasma to the runaway condition if certain circumstances are fulfilled.

To elaborate on the observed phenomena, the different processes which occur must be addressed. Introducing a 5% oxygen flow to the Ar discharge can partially oxidize the target surface. Formation of a partial oxide layer on the target surface (suboxide mode) causes a lower $\gamma_{SE}$\(^{36}\) and consequently, fewer ionization events occur in the vicinity of the target (fewer ions) and a lower discharge current is observed (lower average power due to the constant voltage). This process leads to a state where the target surface oxidizes fully (oxide mode), and a new high $\gamma_{SE}$ takes over due to the presence of high energy impacting ions. Then, the discharge current starts to rise and a significant electron release from the target increases the number of ionization events, i.e. more ions, including metal ions and reactive gas ions, are attracted back to the target and contribute to the discharge current. This increased current raises the sputtering rate and more neutrals have the chance to be ionized and participate in
the discharge current. This process leads to a runaway of plasma to higher current ($\bar{I} > 1$) mainly due to the increased $\gamma_{SE}$. High energy ions (high applied voltage) is necessary in order to sustain the plasma, otherwise more oxidation leads to a very low discharge current (low $\gamma_{SE}$ for low energy regimes) where plasma is not able to be self-sustained. This runaway cannot be sustained. As soon as the plasma reaches a high density, an elevated sputtering rate causes the target surface to be etched more efficiently and thus takes the target to suboxide mode where a reduced $\gamma_{SE}$ violates the criterion of runaway ($\bar{I} < 1$). The same processes as described above repeats, continuing the discharge with a periodic runaway to higher current (see FIG. 2(b)). This process can be understood in the context of oscillating $\gamma_{SE}$ corresponding to different compositions of the target, from metal mode through the suboxide mode to fully oxide mode. A periodic runaway of this kind has not been reported before. It can only be observed if the capacitance network of the power supply is large enough to hold the target voltage constant for the entire pulse length during the runaway.2

In order to estimate the maximum current which can be achieved, one can assume that all the background gas atoms are ionized and contributed to the total current discharging through the target. The current density corresponding to a flow of ions is given by $j_i = QeV_s/n_i$ where $Q$ is the charge state and $v_i$ is the drift velocity. Assuming a Maxwell distribution as a good approximation for bulk plasma, one can consider the gas velocity toward the target to be limited by the thermal repelling of the background gas and to be: $v_g = (2kT_g/\pi m_g)^{1/2}$. Also considering gas ions accelerating through the target sheath according to the target potential $V_t$, one can write the acceleration of a singly charged Ar gas ion (for a collisionless transport over the sheath) as: $a_i = QeV_t/m_g$ and assuming the sheath thickness to be of the order of Debye length ($\Delta x_{sh} = 10^{-1} \text{m}$):

$$2a_i\Delta x_{sh} = v_i^2 - v_g^2,$$

Substituting $a_i$ and $v_g$ using the above equations results:

$$v_i = \left( \frac{2}{m_g} (QeV_s\Delta x_{sh} + \frac{kT_g}{\pi}) \right)^{1/2}.$$

Hence the gas ion current density can be written as:20

$$j_i = Qe\left( \frac{2}{m_g} (QeV_s\Delta x_{sh} + \frac{kT_g}{\pi}) \right)^{1/2} \frac{p_g}{kT_g},$$

where one can consider $Q = 1$, $e = 1.602 \times 10^{-19}$ A s, $m_{Ar} = 40 \times 1.66 \times 10^{-27}$ kg (due to small amount of O, it can be neglected), $k = 1.38 \times 10^{-23}$ J K$^{-1}$, $T_g = 300$ K, $V_t = 700$ V, and $p_g = 0.7$ Pa for this experiment. From this the maximum ion current density can be determined to be $j_i \approx 16600$ A m$^{-2}$. The total ion current can thus be calculated over the effective target area (race-track) of $A_{eff} \approx 24$ cm$^2$ for a 75 mm diameter target as $I_{j(i)} \approx j_iA_{eff}$, which yields $I = I_{j(i)} + I_{SE(i)} = (1 + \gamma_{SE(i)}\gamma_{SE(i)} \approx 1.1j_iA_{eff} \approx 44$ A (a value of $\gamma_{SE(i)}$ is assumed, typical for materials with high secondary electron emission coefficients). Although extreme values have been assumed for the discharge which have excluded the rarefaction and assumed the total background gas atoms to be ionized, the maximum current flowing through the target is about 45 A which is similar to what has been observed during the runaway (see FIG. 3). Thus one can conclude that the emission of secondary electrons is the only parameter which can in principle change to a higher value, cause almost the total gas nearby the target to be ionized, and lead to an increase in the discharge current. It is important to mention that the contribution of self-sputtering to the discharge current is minimal since the sputter yield of the fully oxidized target is very low and the flux of the target materials is too low to initiate a self-sputtering process. It should be also noted that the gas recycling cannot be considered as the main mechanism for the runaway considering the periodic nature of this phenomenon, as in that case the runaway would occur continuously rather than periodically with constant period.

The evolution of the discharge current over the runaway is shown in FIG. 3 for a pulse repetition frequency of 200 Hz. As seen in the figure, the discharge current can increase fourfold (for this specific pulsing frequency, it can be higher for lower frequencies) during the runaway compared to the current out of the runaway. This increased current is higher for lower frequencies, where longer
FIG. 3. The evolution of discharge current around the average power peak. Discharge runaway to a higher density occurs for a few seconds and causes a drastic increase in discharge current. Inset: Top: The average power of an unstable reactive discharge over the runaway is plotted as a function of time. Each point shown in the inset corresponds to a relative current waveform in the main figure. Bottom: The discharge voltage waveforms over a runaway of discharge.

FIG. 4. (a) The maximum and minimum average power of the Ar/5%-O₂ discharge during the oscillation and (b) the period of the oscillations versus pulse repetition frequencies ranging from 140 to 400 Hz. The oscillations repeat several times and the average values and final values of period are plotted for each pulse repetition frequency.
off-time between the pulses results in more oxidation and less etching at the target surface. Hence, a more intense and faster runaway takes place at lower pulse repetition frequencies, which decays to a more moderate and slower runaway for higher frequencies until the number of pulses is enough to establish a compromise between oxidation and etching events. It has to be pointed out that the discharge current gradually rises up sooner compared to the pulse initiation when the target transitions from metal mode to fully oxide mode (runaway), which differs from what has been reported before for the ‘runaway’ in reactive discharges.\\n
An enlarged part of this oscillation is shown in the inset of FIG. 2(b) at 200 Hz revealing that the runaways are periodic. The period ($\Delta T$) and the maximum ($P_{\text{high}}$) and minimum ($P_{\text{low}}$) levels of the average power are plotted as a function of pulse repetition frequency in FIG. 4. $P_{\text{high}}$ drops with increased pulse repetition frequency while $P_{\text{low}}$ rises with increasing frequency until they converge at 400 Hz (stable plasma). The period of runaways increases with increased pulse repetition frequency. This is attributed to the decreased time available between pulses to sufficiently oxidize the area of the target to initiate the runaway. In other words, due to the constant voltage during the on-time of the pulse, the ratio of the highest ($P_{\text{high}}$) and lowest ($P_{\text{low}}$) average power for a certain frequency is equal to the ratio of the total current at the highest ($I_{\text{high}}$) and lowest ($I_{\text{low}}$) average power. Using Eq. (2) and considering a similar ion current for both cases, one can derive:

$$\frac{P_{\text{high}}}{P_{\text{low}}} = \frac{I_{\text{high}}}{I_{\text{low}}} = 1 + \gamma_{SE_{\text{high}}} \frac{1}{1 + \gamma_{SE_{\text{low}}}} \propto \frac{1}{f},$$

(6)

$$\gamma_{SE_{\text{high}}} = \left( \frac{I_{\text{high}}}{I_{\text{low}}} - 1 \right) + \left( \frac{I_{\text{high}} \gamma_{SE_{\text{low}}}}{I_{\text{low}}} \right),$$

(7)

where $f$ is the pulsing frequency and $\gamma_{SE_{\text{high}}}$ and $\gamma_{SE_{\text{low}}}$ are the secondary electron emission coefficients for the highest (oxide mode) and lowest (metal mode) average powers. Hence, it can be suggested that for lower frequencies the fluctuation range of $\gamma_{SE}$ is higher and faster and a more intense runaway occurs whereas this fluctuation range tends to zero when the frequency becomes greater (see FIG. 4(a)).

### IV. CONCLUSIONS

In summary, increasing levels of O$_2$ flows have been introduced stepwise to a HiPIMS Ar discharge of a Ti target in order to investigate the effect of target poisoning on the discharge behavior. The dynamic emission of secondary electrons resulting from a crossover between metallic and oxide modes of the target provides the conditions (high $\gamma_{SE}$ for an oxidized target) for a periodic runaway of plasma to higher density to occur. Massive attraction of positive ions containing mainly reactive gas ions and partly metal ions during the runaway increases the sputtering rate, etches the target surface more efficiently, and takes it from a fully oxide mode to a suboxide mode with reduced $\gamma_{SE}$. The periodic runaway can happen only if the energy of the impacting ions are above 300 eV, enabling $\gamma_{SE}$ to reach a higher value when the fully oxide mode target is achieved, thus initiating the runaway process. The frequency dependence of the periodic runaway further substantiates the emission of secondary electrons to be the main cause of this phenomenon.

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