RAPID RESEARCH LETTER

Reactive process and hysteresis effect in magnetron with magnetized hollow cathode enhanced target

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Abstract
The hysteresis effect in the reactive process was investigated in the magnetron with a magnetized hollow cathode enhanced target (HoCET) in which the target is coupled with the hollow cathode magnetized by the magnetic field of the magnetron. The process, where both the magnetron and hollow cathode plasmas are combined, is compared to the magnetron sputtering. The hysteresis curve in the magnetized HoCET magnetron, recording the titanium emission intensity versus varying content of nitrogen in the gas mixture exhibits a local maximum on the increasing part of the curve. The hysteresis curve is shifted to lower contents of nitrogen than the hysteresis curve for the magnetron. It is concluded that more efficient utilization of the reactive gas takes place in this device.

KEYWORDS
hollow cathode enhanced target, ionized magnetron, magnetized hollow cathode, reactive process in ionized magnetron

1 INTRODUCTION

Hollow cathodes do not exhibit a hysteresis effect in the reactive process,[1] Processing points, recording the sputtering (or sputtering + evaporation) rate by optical emission from the target metal follow the same curve both for increase and decrease of the content of the reactive gas in the reactive gas mixture with argon. This was proved even for target areas comparable with areas of the magnetron targets. The only hysteresis observed in the hollow cathode was connected with the hysteresis of target temperature at certain regimes.[2] Magnetron with magnetized hollow cathode enhanced target (HoCET) is the magnetron coupled with the magnetized hollow cathode utilizing the magnetic field of the magnetron, where magnetron target as one plate of the hollow cathode is paired with the additional circumferential parallel plate. The HoCET magnetron exhibits higher deposition rates than the magnetron.[3] The device has recently been tested also for the reactive deposition of TiN and compared to the magnetron deposition.[4] The HoCET magnetron provided a 140% increase in the TiN reactive deposition rate over the non-reactive deposition of Ti, at 14 mTorr (1.87 Pa). Even stoichiometric films were obtained at deposition rates higher than the deposition rate of Ti. The rate increase was found in the interval of 1–5% N2 in the gas mixture, with a maximum at 2.4%. The increase coincided with the increased production of Ti and Ti+ and increased ionization of Ar. Coupling between the pressures and the optimum nitrogen contents where the maximum ionization and maximum deposition rates occur was observed.

Thus, the HoCET source with respect to deposition rates and properties of the deposited compound resembles the hollow cathodes. However, it was not clear if hysteresis plays a role in this source and to what degree the influence of the magnetron is reflected in the HoCET device behaviour. This paper describes the reactive process and compares both sources.
2 | EXPERIMENTAL

Details about the HoCET concept have been already published elsewhere. A schematic sketch is shown in Figure 1. In experiments, a planar magnetron ONYX-4MAGII produced by Angstrom Sciences Inc. with a circular Ti target (diameter of 4 inches) was used. The circumferential hollow cathode plates were also manufactured from Ti. The multiple gas inlets were arranged inside the slit distributing the gas between the cathode plates. The total gas flow was 60 sccm, the pressure in the chamber was 14 mTorr. The 13.56 MHz radiofrequency (rf) power supply providing a delivered power of 1.2 kW was applied in the experiments. The self-bias voltage $V_{s-b}$ was measured at the powered electrode.

The optical emission from the plasma was recorded by a PLASCALC-2000-UV–VIS–NIR Plasma Monitoring and Process Control System. Emission spectra were collected through a side window by optic fibre. Optical emissions from Ti ($\lambda = 3,653.5$ Å), Ti$^+$ ($\lambda = 3,685.2$ Å), Ar ($\lambda = 4,158.6$ Å), Ar$^+$ ($\lambda = 4,103.9$ Å), and from N$_2$ (second positive system) and N$_2^+$ (first negative system) were used for the optical emission spectroscopy (OES) analysis.

3 | RESULTS

Figure 2 shows a typical hysteresis curve where the sputtering rate is recorded as an optical emission intensity from Ti and Ti$^+$ in the magnetron. The nitrogen content increase and subsequent decrease are indicated by arrows. The film consumption limit given by the arrival of the gas manifests itself up to 8% of the nitrogen in the mixture with argon. The hysteresis effect is associated with the necessity to cleanse the target during a successive decrease of nitrogen content in

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**FIGURE 1** Schematic of the magnetron with magnetized hollow cathode enhanced target

**FIGURE 2** ($\text{Ti + Ti}^+$) optical emission intensity versus nitrogen content in the mixture with argon. Magnetron discharge
the gas mixture. In Figure 3, there is the same process plotted as \(V_{s-b}\) versus \(N_2\). This graph features a picture very similar to the hysteresis curves recorded for partial pressures versus \(N_2\), see example.\(^{[7]}\) The relation between \(V_{s-b}\) and partial pressure of the reactive gas is clear – by increasing partial pressure of nitrogen, the space-charge sheath thickness at the powered electrode increases, and the self-bias voltage increases. The hysteresis effect is observed in the same section of \(N_2\) content as in Figure 2.

Photographs in Figure 4 show the discharge evolution in the magnetron with magnetized HoCET in the reactive mode with varying nitrogen content in the mixture. Figure 5 features a more complicated hysteresis effect in the magnetron with magnetized HoCET. Note that this is a representative curve, based on many measurements. The curve features a maximum at 2% of the nitrogen in the gas mixture at the increasing nitrogen content part of the curve. This is in agreement with our previous measurements where the maximum production of Ti and Ti\(^+\) species and maximum deposition rates were achieved.\(^{[4]}\)

We can see the hysteresis again, but the whole hysteresis area is shifted to the left to lower nitrogen contents as compared to the magnetron. The same effect can be seen in Figure 6 for \(V_{s-b}\) versus nitrogen content dependence. This behaviour signals that there is a sufficient amount of activated reactive gas available “earlier” than in the magnetron, for the transfer to the reactive mode, even though the amount of the delivered reactive gas is actually lower. Sufficient arrival of the “activated reactive” gas was also confirmed by the results in ref. \(^{[4]}\), where both high deposition rates of TiN and even stoichiometric films were achieved at low nitrogen percentages. The authors believe that a higher degree of activation of nitrogen, either increase of \(N_2\) excitation or increase of \(N_2\) molecule ionization occurs. The experiment described here takes place at rather low power, only in the presence of the hollow cathode discharge, not of the hollow cathode arc (HCA). Therefore, the emissions from \(N_2\) and \(N_2^+\) have not been observed at the \(N_2\) contents below 10%. In the HCA regimes, excited \(N_2\) and ionized \(N_2^+\) are usually observed also at very low concentrations of \(N_2\), for example at 0.5% in ref. \(^{[8]}\) The emission from \(N_2\) and \(N_2^+\) were collected and analysed both for the HoCET and magnetron. The measured ratios of emission intensities from \(N_2^+\) and Ti and also ratios of emission intensities from \(N_2^+\) and (Ti + Ti\(^+\)) were always higher for the HoCET than for the magnetron, the increase factor being approximately 1.3. Z. Pang and coauthors claim\(^{[9]}\) that the ratio of \(N\) to Ti atoms in the film, \([N]/[Ti]\), is linearly related to the ratio of emission intensities from \(N_2^+\) and Ti, \(I(N_2^+)/I(Ti)\). The results indicate more efficient incorporation of nitrogen into the TiN film for the HoCET.

Another indirect proof that in the “earlier” section of the curve there exist excited and ionized \(N_2\), is the behaviour of the emission from Ar. The decrease of emission from Ar appears at lower \(N_2\) content than in magnetron, its onset is also shifted to the left. In the study on optical emission from magnetron discharges as a function of a composition of argon-nitrogen mixtures,\(^{[10]}\) the authors analysed how the signals from \(N_2\) and \(N_2^+\) and signal from Ar evolve mutually. With increasing emissions from \(N_2\) and \(N_2^+\), the Ar signal continuously decreases. Figures 7 and 8 compare the Ar emissions in the magnetron and in the HoCET magnetron. While the emission intensity in magnetron remains high up
FIGURE 4  Hollow cathode enhanced target (HoCET) magnetron discharges for 0, 2.44, 4.11, 6.25, and 100% of $N_2$ in the mixture with Ar

... to approximately 8% of $N_2$, the signal starts to drop almost immediately with the admission of $N_2$ to the gas mixture in the HoCET magnetron. It can be, therefore, suggested that the earlier reduction of the Ar emission really indicates the presence of activated nitrogen.

4 | CONCLUSIONS

The reactive process in the magnetron with magnetized HoCET features a hysteresis curve that reflects the presence of the hollow cathode. Compared to the reactive process in the magnetron, the hysteresis curve in the HoCET magnetron...
FIGURE 5  (Ti + Ti⁺) optical emission intensity versus nitrogen content in the mixture with argon. Magnetized hollow cathode enhanced target magnetron discharge

FIGURE 6  Self-bias voltage versus nitrogen content in the mixture with argon. Magnetized hollow cathode enhanced target magnetron discharge

exhibits a maximum on the increasing N₂ part of the curve. The whole hysteresis curve is shifted towards lower contents of N₂ in the mixture with argon than in the magnetron. It is believed that this shift is a result of a more efficient activation of the reactive gas (nitrogen), and thereby more effective utilization of the reactive gas in the magnetron with magnetized HoCET and its incorporation into the growing TiN film.

Experimental results obtained in this work confirm that a concept of combination of the magnetron and hollow cathode plasmas using a circumferential magnetized hollow cathode arranged at the magnetron target (HoCET) magnetized by the magnetron magnetic field considerably improves the performance of the magnetron sputtering. The concept is, therefore, very promising for applications where both a high deposition rate and effective incorporation of the reactive gas into the film, which affects the film properties, are requested.
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