TRANSITION STAGES IN THE REACTIVE MAGNETRON DISCHARGE

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Abstract

A phenomenon of enhancing and diminishing of the hysteresis loop in the current - voltage characteristics with time rate of change of discharge current (dI/dt) was studied. A good correlation was found between the jumps in the hysteresis loops and time evolution of plasma parameters after discharge transition between two different stages (each of them characterised by a constant intensity of the discharge current). A phenomenological model is proposed based on the surface processes at the target.

1. Introduction

The reactive magnetron discharge shows rather often hysteresis phenomena in the current - voltage characteristics, in dependence of other physical parameter such as partial pressure of the gas components or total gas pressure versus e.g gas flow rate of the reactive gas or discharge current intensity [1]. In a previous paper [2] we found that the current - voltage characteristics can be used for monitoring a reactive process because the magnetron discharge can be in a reactive or a metallic mode for different discharge currents and a given ratio of the partial pressure of buffer gas to reactive one [2]. Recently, dependence of the hysteresis in the current - voltage characteristics versus rate of change of the discharge voltage has been reported and a sort of resonant phenomenon was identified [3,4].

In the present paper, experimental results are presented on a similar reactive magnetron discharge but following the time evolution of some plasma parameters when discharge current intensity is controlled so the discharge current was either a saw-tooth or a square pulse with different rates of change. Under the assumption that at constant discharge current intensity any change in the discharge potential corresponds to a change of the impedance of the plasma - surface system, a phenomenon of enhancing and diminishing of the hysteresis loop in the current - voltage characteristics with the time rate of change of the discharge current (*dI/dt*) was studied. Gas adsorption and its chemical reactions with the sputtered metal at the surfaces seem to be responsible for the time behaviour of plasma impedance [4,5,6]. A phenomenological model is proposed based on elementary processes taking place at the target surface as a competition between sputtering and forming of chemical compounds.

2. Experimental set-up

A stainless steel discharge chamber 20 cm in diameter and 25 cm in length was pumped down to about 10^{-5} Torr with a standard oil-diffusion pump provided with a freon trap. The argon or argon - reactive gas mixture, at a total pressure ranging between 10^{-3} and 5.0×10^{-3} torr, were continuously introduced into the discharge chamber through mass flow rate controllers with two ways under the conditions of mass flow rate less than 10 sccm. The magnetron structure is a standard one with annular permanent magnets (35 mT at the cathode's surface). Disc-shaped Ti, TiAl or Cu targets (2 mm thick and 105 mm in diameter) were used. The magnetron was placed close to the center of the discharge chamber.

Time evolution of the discharge current intensity for different *dI/dt* values and of the partial pressure of the reactive gas component and the main ionic species were registered using a computer-controlled d.c. power supply (operated in a constant-current mode), a data acquisition system and a residual gas/plasma monitoring system (Hyden Analytical). The orifice of the gas/plasma analyser was at 70 mm from the erosion groove underneath the magnetron plasma ring. The measurements were done for different constant mass flow rates of inert and reactive gas (argon - oxygen, argon - air for Cu target and argon - nitrogen, argon - oxygen, argon - air for Ti and TiAl target) between 10^{-3}-5.10^{-3} Torr.

3. Experimental results and discussion

When the discharge current intensity is varied a transition between the *reactive mode (RM)* and *metallic mode (MM)* (for Ti, TiAl and Cu targets) can be obtained and the hysteresis in the current-voltage characteristics can be registered. Figure 1 shows a typical *I - V* hysteresis curve registered with a saw-tooth variation of the discharge current intensity (Ti target in air, $Q_{air} = 0.3$ sccm, $Q_{Ar} = 1.7$ sccm and $dI/dt = 0.015$ A/s).

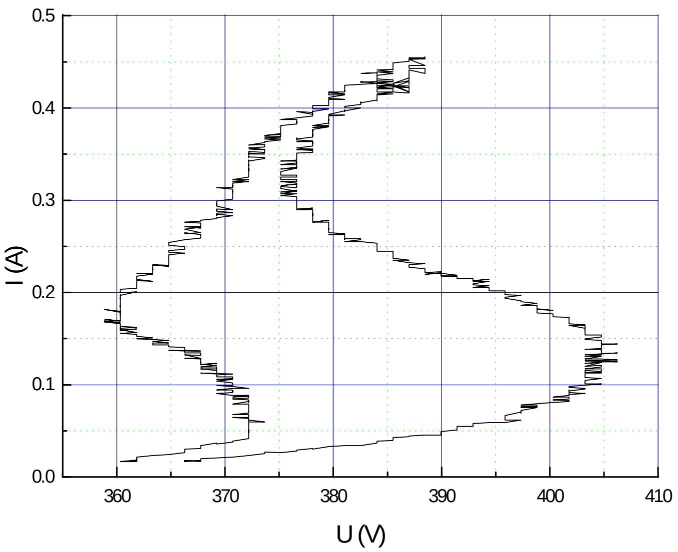

Fig. 1. Hysteresis effect in I - U characteristics

When *I* increases, the first transition (from *RM* to the *transition mode-TM*) occurs at a threshold value I_{TI}= 0.14 A while a second one (from *TM* to *MM*) occurs at I_{MI} = 0.3 A. When I decreases the first transition (from *MM* to *IM*) occurs at I_{MD} = 0.18 A and the second one (from *IM* to *RM*) occurs at I_{TD} =0.05 A. The width of this hysteresis loop is 45 V. The discharge voltage *V*, which reflects both the surface phenomena at the cathode and the volume plasma processes, gradually increases until first transition takes place. After this transition the discharge voltage decreases with increasing of the discharge current, and, finally, starts increasing again in *MM.*

The decrease of discharge voltage in the *TM* can be explained as follows. In reactive sputtering of materials, the main processes occurring at the cathode surface are chemical reactions and sputtering. For low discharge currents the cathode is covered by compounds (TiN, TiO_2) while for high discharge currents the cathode surface becomes metallic. Since the discharge current intensity has a radial distribution (the surface current density is maximum where the magnetic fields lines are parallel to the target surface and decays to zero on both sides there where the magnetic fields lines are perpendicular to the surface), there is a difference in sputtering rate between different cathode surface locations. So, for $I > I_{Ti}$, there is a central region on the ring cathode surface where the surface becomes metallic and the rest of it is covered by the compound. One can consider that in the *TM* there are two discharges in parallel: one with a metallic cathode and another one with a compound cathode. The surface of metallic zone gradually increases in time until it becomes equal to the overall cathode surface; at this moment, the discharge passes in *MM*. The impedance of discharge is given by two impedances coupled in parallel: one with high value (for the discharge with compound cathode) and another one with a lower value (for the discharge with metallic cathode). Since the impedances are proportional to the reverse of the areas of the surfaces, and these areas vary in time, in *TM* the. potential passes from a higher value (in *RM*) to a lower one (in *MM*). For discharge current intensities lower than I_{TI} the hysteresis does not occur as the cathode remains all the time in a reactive mode (there is a compound layer on the cathode surface and the compound rate formation is greater than the sputtering rate. In this case, the *I - V* characteristics are independent on *dI/dt.*

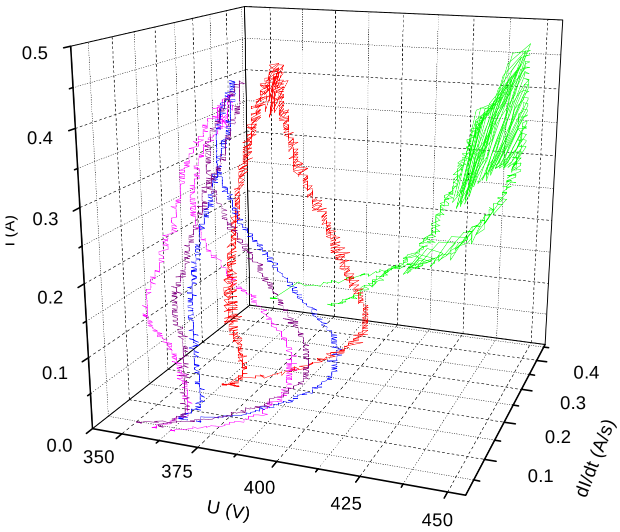

Fig. 2. I - U characteristics as a function of dI/dt

Fig. 2 shows the influence of *dI/dt* on hysteresis (Q_{Ar}= 1.7 sccm,Q_{air} = 0.3 sccm). Curves are similar to those registered for time variation of discharge voltage in argon-nitrogen with Ti cathode [4]. As *dI/dt* increases, the hysteresis width first increases and then descreases while the transition point from *RM* to *MM* shifts to a higher values of *I* (the time variation is lower than the time necessary to clean-up the cathode surface) while the transition point from *MM* to *RM* shifts to lower values (the variation time is lower than the time necessary to cover the cathode's surface with compound).

The time dependences of the concentration of main ion species present in a reactive magnetron discharge with Ti cathode, under the same conditions, obtained by mass spectrometry, are presented in Fig. 3.

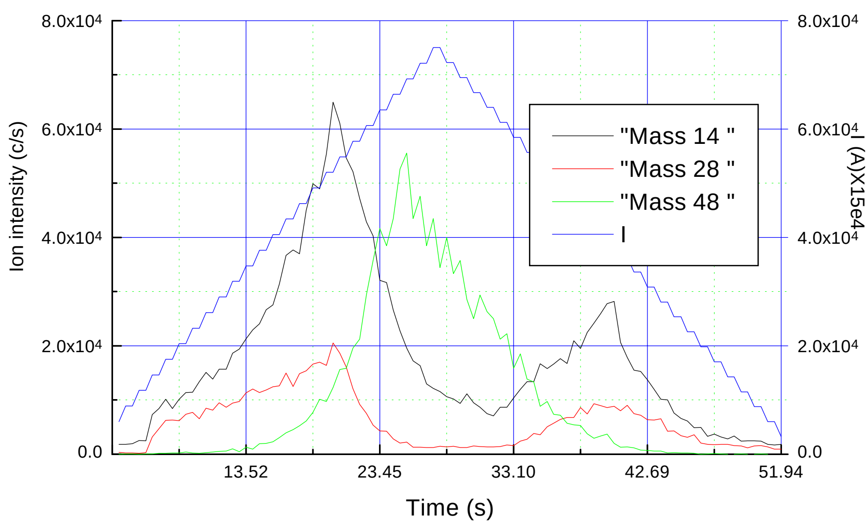

Fig. 3. Time variation of ion species

Important changes in the intensities of signals are observed for different dI/dt values. Firstly, the reactive ion intensities increase for $I<I_{TI}$, then decreases between I_{TI} and I_{MM}. The increase is due to the increase of the dissociation rate of the reactive gas connected with the increase of the discharge current; the decrease is due to reactive gas consumption on behalf of Ti atoms which occurs in a great number when $I > I_{TI}$.

Gas adsorption and compound formation by chemical reactions with the cathode material, sputtering of this compound and of cathode material, as well as chemical reactions at the surfaces in contact with magnetron plasma seem to be responsible competing factors for the observed time behaviour of plasma impedance [4]. To prove these assumptions, measurements are under progress on similar reactive magnetron discharges, but following the time evolution of discharge potential after discharge transition between different stages (each of them characterised by a constant intensity of the discharge current).

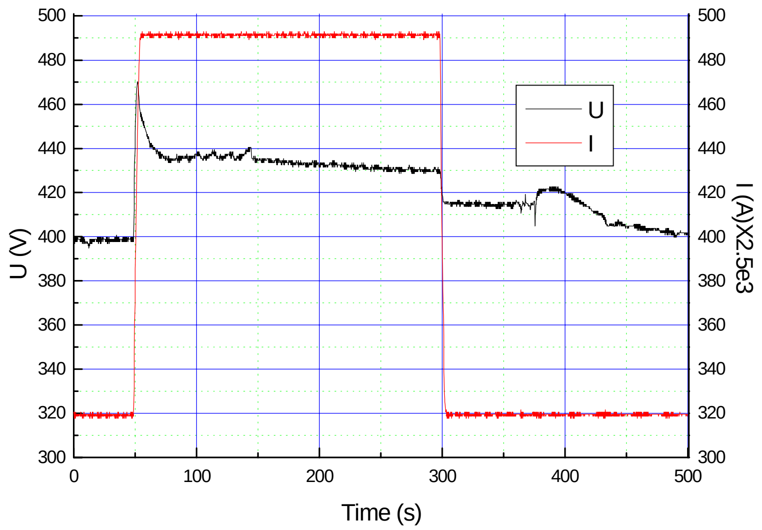

Fig. 4. Potential variation for a square pulse discharge current

The transition between these two values of the discharge current (Fig. 4) is a square pulse with a variation between I_{min}= 0.02 A and I_{max}= 0.2 A (Q_{air} =0.5 sccm, Q_{Ar} = 1.7 sccm). This plot clearly shows that the discharge voltage can be used to monitor the time evolution of the cathode processes related to the formation and sputtering of compound at its surface.

4. Conclusions

Hysteresis behaviour and the effects on it of the *dI/dt* have been investigated and their influence on hysteresis process were discussed. The results explain the important connection between *dI/dt* and hysteresis. As *dI/dt* increases, the hysteresis width increases then decreases, the transition point from *RM* to *MM* shifts to higher values of *I* while the transition point from *MM* to *RM* mode shifts to lower values. The change in transition points as a function of *dI/dt* were explained taking into consideration time dependent reactive product formation and sputter off balance. A phenomenological model is proposed on the basis of the coverage - dependent discharge impedance considering that in the *TM* there are two discharges in parallel: one with a metallic cathode and another one with a compound cathode. We have found that when a reactive magnetron discharge is powered by a constant current source the *I - V* characteristics are dependent on *dI/dt* and when these characteristics are used for reactive process monitoring the rate of change of discharge current must be specified in order to achieve reproducible results.

References

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