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Transitory phenomena in pulsed reactive magnetron discharge

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The paper presents the experimental results obtained in the study of transitory phenomena and hysteresis effect for a magnetron discharge in reactive-noble gas mixture, N_2/Ar, with titanium target. The range of pressure was between 3 mTorr and 15 mTorr. The hysteresis effect is made evident in the current-voltage characteristic obtained by increasing, followed by decreasing, with constant rate, of discharge current and recording the voltage between cathode and anode. The transitory phenomena were studied both electrically and spectrally when the discharge current is switched as a step function between two constant values. As an electrical parameter was studied the voltage between cathode and anode, and as spectral parameter was measured intensity of following spectral lines: Ar^+ (420,06 nm), Ti^+ (503,7 nm) and N_2^+ (358,2 nm). These parameters have different temporal evolutions with the current discharge increasing as compared with the current discharge decreasing. This dissymmetry provides information about the rate of the processes occurring at the cathode during the formation of compounds on the surface, respectively, during the sputtering.

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Keywords: Hysteresis, Magnetron discharge, Transitory phenomena

1. Introduction

The reactive magnetron is a magnetron discharge where the operation gas is a mixture including constituents that may chemically react to the target metal. The reaction may occur both directly, either on the cathode surface or on any surface that the sputtered material is laying, and in the plasma volume. Under these circumstances the micro and macro parameters of the discharge may evolve in time according to the reaction velocity of the formation and respectively destruction of some compounds.

The hysteresis effect is brought about by changing conditions of the cathode surface, there is a competition between the formation of compound by physical and chemical absorption of the reactive gas on the cathode surface, and the sputtering of the compound. This competition depends on the rate of the metallic atoms sputtered, the ratio of the rate inert gas, reactive gas (Ar/N_2), and the time rate of the change of the discharge current being related to the chemical reactions in both active surface and plasma volume.

The analysis of the phenomena related to the relaxion processes which follow the step transition between two regimes, each of them being characterized by constant discharge current intensity is the object of investigation of the transitory phenomena.

In the case of magnetron discharge with a titanium target and a nitrogen-argon mixture as a reactive gas, the discharge impedance under metallic regime is lower than under compound one. It is due to the fact that the electronic emission to the cathode – by the processes γ-Townsend – is lower with the titanium nitride than with the metallic titanium.

2. Experimental setup

The experimental apparatus is shown in Fig. 1. The vacuum chamber was depressurized by a oil-diffusion pump to a lower pressure of about 1×10^{-6} Torr [1]. The magnetic field measured at the target surface was 80 mT. The magnetron was operated in a d.c. mode using a power supply that may be controlled by a pulse generator. The operation gas was a mixture of argon and nitrogen controlled by two mas flow controllers. The spectral parameter was studied using a Carl Zeiss monochromator and the optical spectra was recorded using an Oriel spectrometer.

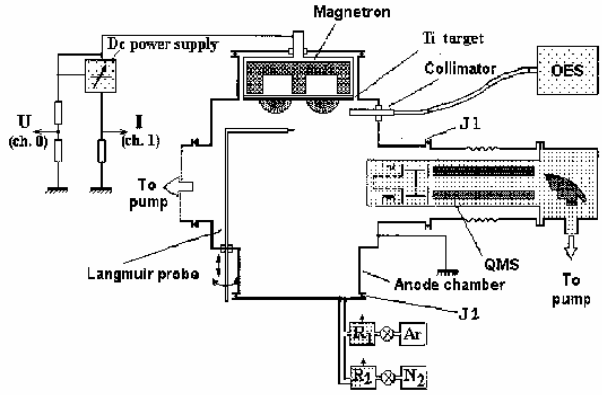


Fig. 1. Experimental setup.

3. Results and discussion

The electrical parameter was studied by measuring the variation of the voltage discharge when rapidly changing

from one steady state discharge featuring a constant value of the discharge current to another whose value was also maintained constant (Fig. 2). The voltage modification on the discharge gives the first information on the variation of the discharge impedance. The electrical measurements have proven that the variation of the voltage is determined generally by the phenomena occurring on the surface of the cathode.

A growth of the discharge current (AB, Fig. 2a) is followed by a similar growth of the discharge voltage (AB, Fig. 2b). The point A (Fig. 2b) corresponds to the situation when the cathode is covered by the compound (titanium nitride). The point B on the voltage characteristic corresponds to the situation when the magnetron operates in compound regime at of the maximum current value. Thereafter, it follows an exponential decline (BB', Fig. 2b) caused by the sputtering process of the nitride on the surface of the titanium target. The discharge voltage is kept constant again while operating with the metallic target (B'C, Fig. 2b). While the discharge rate decreases up to its initial value (*CD*, Fig. 2a), the burning voltage does not reach immediately the value corresponding to the level *A* (Fig. 2b) but decreases up to an intermediate value *D'*. It undergoes afterwards a more intricate evolution before recovering its level as previously mentioned (*DD'*, Fig. 2b). The electrical and spectral methods have proven that this segment is also related to the phenomena occurring on the surface of the target and especially to the formation of the compounds on its surface. The discharge impedance depends on the plasma parameter and the electrical properties of the thin layer on the cathode and anode.

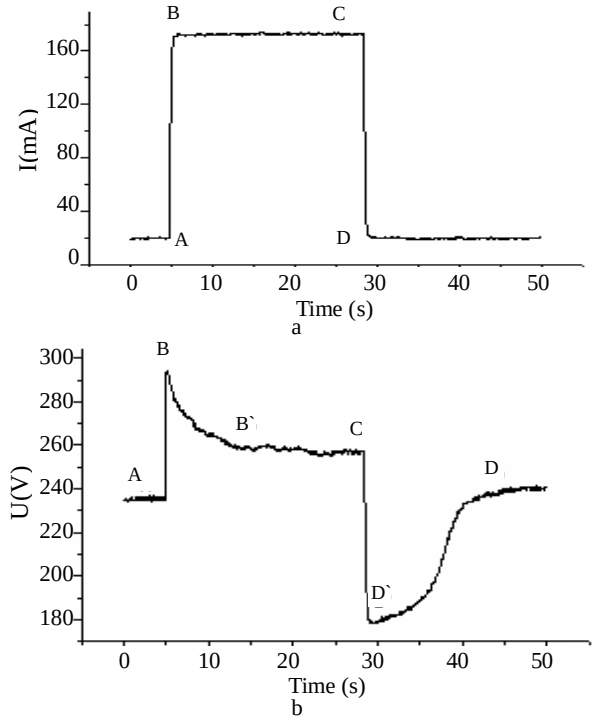


Fig. 2. Time evolution of the discharge voltage of a plane magnetron discharge with titanium target (b) corresponding to the discharge current (a), p = 10 mTorr, Q(Ar) = 12 sccm, Q(N_2) =0.5 sccm;

Intensity of spectral line Ti^+ (503,7 nm) for different pulses of the discharge current was measured as spectral parameter. In Fig. 3 both the evolution of the discharge voltage and the intensity of spectral line of Ti^+ (503.7 nm) corresponding to the discharge current are shown. The relaxation of the discharge voltage depends on the amplitude of the current pulse. The time of the sputtering process of the nitride on the surface of the titanium target increases with the decrease of the maximum current value, for the same value of the ratio of the mass flow rates of both inert gas and reactive gas (Ar/N_2), respectively. In Fig. 3c, the relaxation time of the intensity of the spectral line of Ti^+ is the same with the time of the relaxation processes after increasing of the discharge current. The intensity of spectral line Ti^+ is maximum when the magnetron discharge operates in metallic regime. Also the value of the intensity of spectral line Ti^+ depends on the maximum current value. By decreasing the intensity of the discharge current, the shape of evolution of the discharge voltage has two slopes, one corresponding to formation of the centres of nucleation on the surface cathode, and one corresponding to the coverage of the surface cathode with a thin nitride layer.

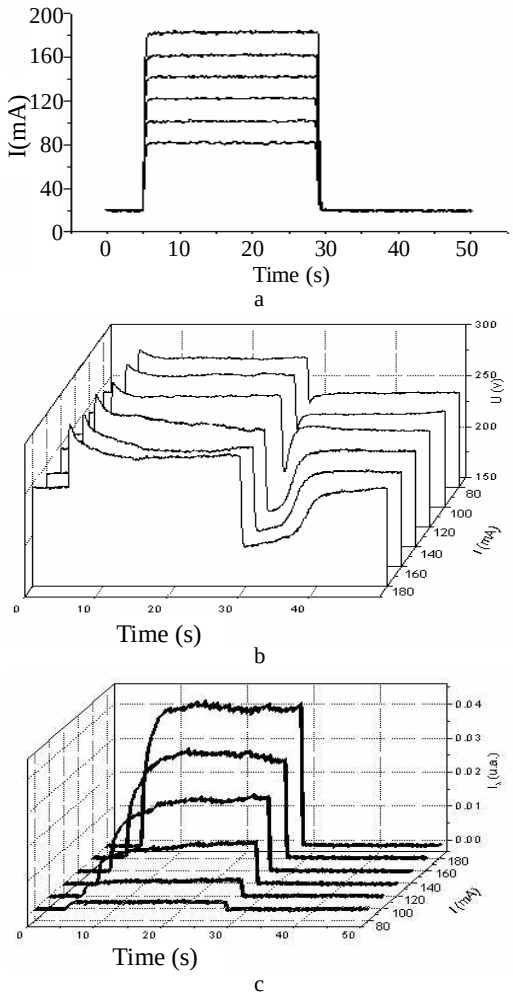
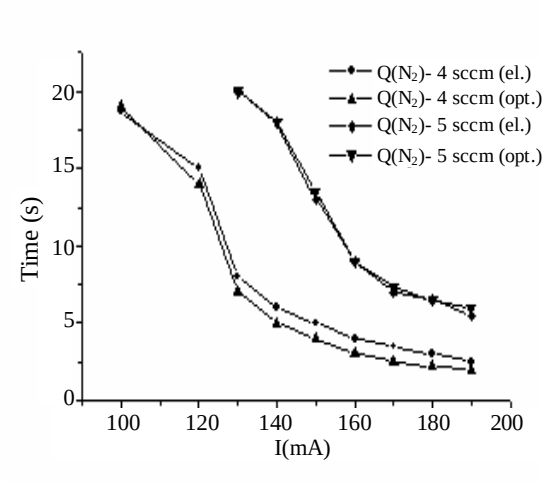
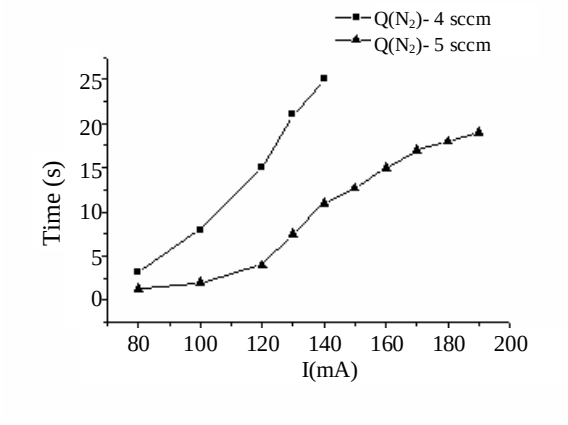


Fig. 3. Time variation of the discharge voltage (b) and the intensity of spectral line of Ti^+ (503.7 nm) (c) corresponding to the discharge current (a), p = 10 mTorr, Q(Ar) = 12 scm, Q(N_2) = 0.4 sccm.

The sputtering time of the compound from the cathode surface measured by an electrical parameter and, respectively, by a spectral one, and time of compound formation on the surface measured also as an electric parameter are shown in Fig. 4. There is a good agreement between the time of sputtering of the compound on the cathode surface measured electrically and the time of sputtering compound on the cathode surface measured spectrally (Fig. 4a).



a



b

Fig. 4. a) The sputtering time of the compound from the cathode surface measured by electrical parameter, respectively, by spectral line intensity and b) time of compound formation on the surface measured by an electric parameter. p = 10 mTorr, Q(Ar) = 12 sccm.

In Fig. 5 the increasing rate of the spectral line intensity (Ti^+, 503.7 nm) is shown versus the value of the maximum of the pulse current for three different values of the mass flow rates of nitrogen gas. The increasing rate of the spectral line intensity corresponds to the rate of the sputtering process.

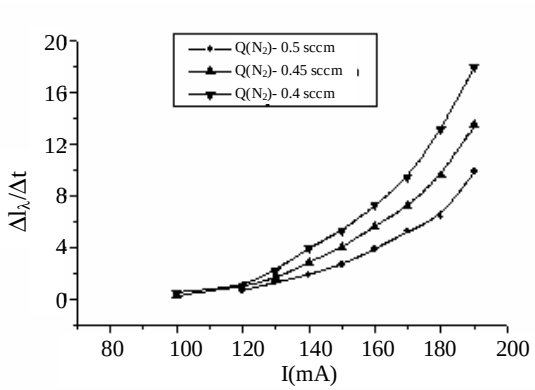
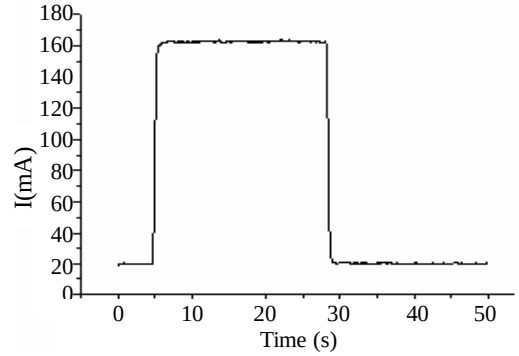
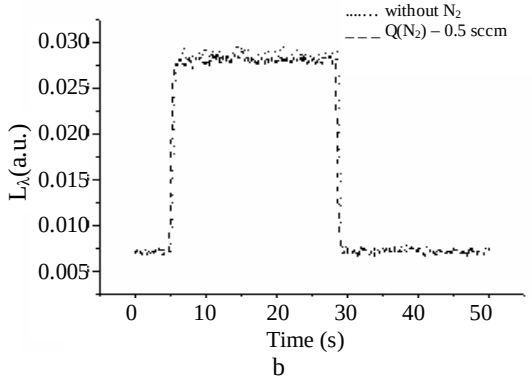


Fig. 5. I The increasing rate of the spectral line intensity of Ti^+ (503.7 nm) versus maximum intensity of the pulse current. p = 10 mTorr, Q(Ar) = 12 sccm.

The spectral line of argon Ar^+ (420.06 nm) was also measured. The growth of the discharge current is accompanied by a similar growth of the intensity of spectral line (Fig. 6). The shape of the intensity of the spectral line is the same with the shape of the discharge current. Furthermore, there is no difference between the intensity of the spectral line measured when discharge operates with nitrogen and when the discharge operates without nitrogen that because the ratio of the mass flow rate of the reactive gas (nitrogen), to the gas mixture (N_2/Ar), is too small.



a



b

Fig. 6. a)Time variation of the spectral line intensity, Ar^+ (420.06 nm) and (b) the time evolution of corresponding discharge current intensity, p = 10 mTorr, Q(Ar) = 12 scm, $Q(N_2)$ = 0.5 sccm.

The time variation of the discharge voltage (b) and the intensity of spectral line of N_2^+ (358.2 nm) (c) corresponding to the discharge current (a), is presented in Fig. 7. The growth of the discharge current is accompanied by a similar growth of the intensity of the spectral line. Then follows an exponential decay caused by the removal of the nitride layer and tran sition to metallic operation regime. When the discharge current intensity decreases to its initial value, the intensity of the spectral line decreases to a minimal value, the magnetron operates in metallic regime and takes place an absorption of the nitrogen on the surface of the cathode. In according with the coverage of the cathode surface with a thin nitride layer, the intensity of the spectral line of N_2^+ increases. The time evolution of the of spectral line of N_2^+ after increasing the discharge current and after decreasing of the discharge current to the initial value (Fig. 7c) follows mainly the evolution of the voltage discharge (Fig. 7b).

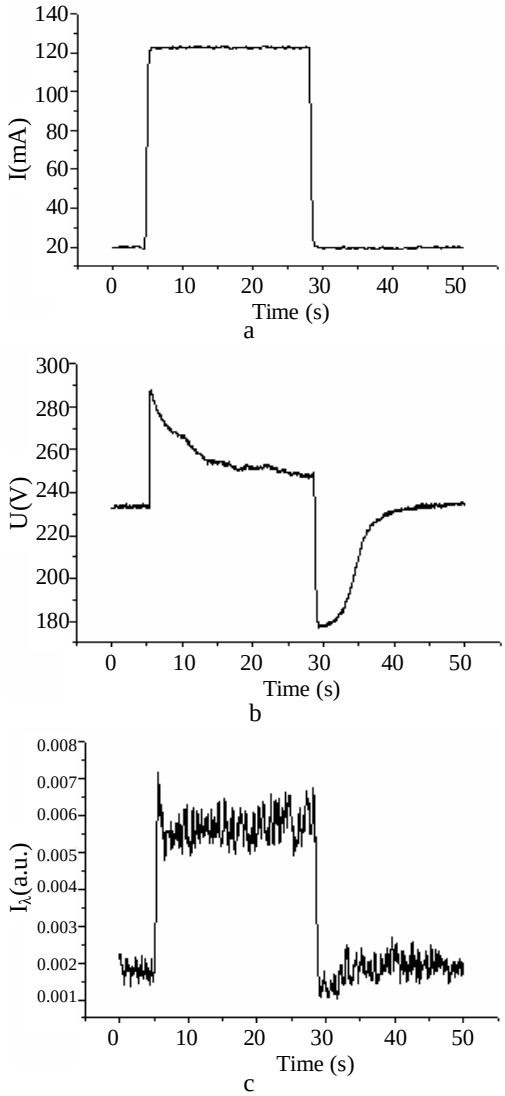


Fig. 7. Time variation of the discharge voltage (b) and the intensity of spectral line of N_2^+ (358.2 nm) (c) corresponding to the discharge current (a), p = 10 mTorr, Q(Ar) = 12 sccm, Q(N_2) = 0.4 sccm;

4. Conclusions

The hysteresis effect can be explained as a result of the competition between sputtering process and formation of compounds at the target surface related to the chang of the parameters in front of the cathode.

The electrical and spectral methods used in this paper are complementary and provides information about the rate of the processes occurring at the cathode during the formation of compounds on the surface, and during the sputtering.

Acknowledgement

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