DOUBLE MAGNETRON CLUSTER SET-UP FOR SYNTHESIS OF MICRO AND NANO STRUCTURE COATINGS

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In the present paper, the results studying the technological regimes of reactive magnetron sputtering in cluster set-up with two planar magnetrons, plasma source and medium energy ion source are presented. Magnetron current-voltage characteristics as well as dependencies of the magnetron current, voltage and the total pressure in the chamber on the reactive gas flow are presented with emphasis on the features of the joint work of the two magnetrons with targets of different materials using different reactive gases. The technological "window" is determined on the basis of the measured characteristics.

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INTRODUCTION

In previous study [1], the results of elaboration and investigation of cluster technological setup for synthesis of complex compound composites were demonstrated. The presented set-up consists of complimentary DC-magnetron system, RF-inductive plasma source and ion source. The set-up system allows to form the fluxes of metal atoms, chemically active particles, and ions independently from each other, as well as to synthesize the thin films of complex compound composites, including nano composites.

The results of studying the different module components were published previously: the research of the low-pressure DC magnetron [2]; the research of arcing processes at the magnetron target in the oxygen atmosphere [3]; the research of the target passivation [4]; the research of the RF inductive plasma source [5].

On the base of this module we designed the experimental multifunctional cluster ion-plasma system with parameters corresponding to the demands of industrial operation. The main purpose of this system is synthesis and processing of complex-composite (including nano-composite) coatings and structures based on $TiAlN_x$, $TiAlO_x$, Al_2O_3, $ZrAlO_x$ and their combinations. Peculiarities of the system operation with reactive gas filling were reported in [6] where the results of measuring the current-voltage characteristics of magnetron discharge with various target materials in argon mixtures with oxygen and nitrogen were presented.

The experiments showed that the magnetron discharge in reactive atmosphere can glow in two modes with hysteretic transition between them. However, these results were insufficient for understanding the double-magnetron system operation in the reactive gas. The present paper describes the results of studying the simultaneous operation of two magnetrons with emphasis on mutual influence of the magnetron discharges on each other. Basing on the obtained results the deposition technology of ZrAlOx coatings is developed.

1. EXPERIMENTAL SETUP

The cluster set-up is schematically shown in the Fig. 1. The system consists of two low-pressure magnetrons 2, 7 located at different sides of the chamber, the RF inductive source of plasma and activated particles of reactive gas 3 located inside the chamber. The relative location of these components is chosen to provide the possibility of the simultaneous action on the processed surface of the flows of metal atoms, activated particles of reactive gas and ions of rare or reactive gas.

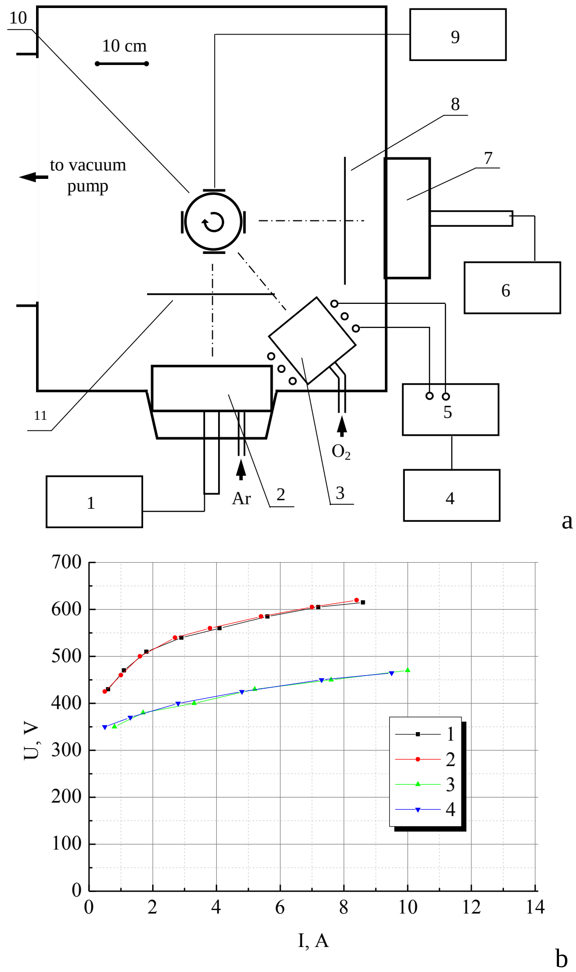


Fig. 1. Scheme of the cluster set-up for complex composite compounds synthesis. 1, 6 – DC magnetron power supply; 2, 7 – magnetron; 3 – RF ICP source; 4 – RF generator; 5 – RF matchbox; 8, 11 – shutter; 9 – pulsed power supply for samples polarization; 10 – samples rotation system

A planar magnetron with permanent magnets is used in the system (Fig. 2). The magnetron power supply allows to bias the magnetron target at up to 1 kV negative potential at the discharge current up to 20 A, maximum power of the supply is 6 kW. The magnetron targets of 170 mm diameter are made of aluminum, zirconium or titanium. Distance from the target to the processed samples is variable within the limits 100…500 mm in the case of pure magnetron deposition, and is fixed in approximately 300 mm for the case of simultaneous operation of the magnetron and the ion source.

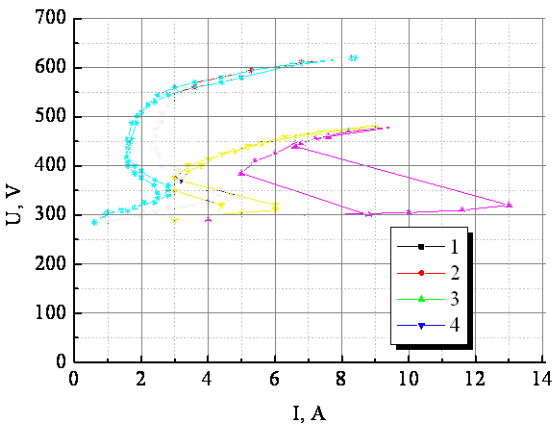


Fig. 2. CVC of two magnetron discharges with Zr (lines 1, 2) and Al (lines 3, 4) targets. a – working gas is pure argon; b – mixture of argon and oxygen. Lines 1 and 3 correspond to single-magnetron operation; lines 2 and 4 show influence of second magnetron. Base argon pressure $P = 1.5 \times 10^{-3}$ *Torr*

2. EXPERIMENTAL RESULTS

The oxide Al_2O_3 and ZrO_2 coating is deposited in high vacuum pumping system with the base pressure about 10^{-5} mBar. There is the problem of target oxidation during deposition process. Under the excessive oxygen flow conditions the process shifts to the target passivation regime. The sputtering process should be made in the regimes far from the target passivation both for aluminum and for zirconium target materials for oxide coatings deposition with highly stoichiometric composition. Such deposition conditions allow also to avoid micro-arcs and micro-drops formation increasing the corrosion resistance.

The problem of micro-arcs and micro-drops formation becomes actual during simultaneous operation of two magnetrons. Fig. 2 presents the current-voltage characteristic (CVC) of the magnetrons with targets of aluminum and zirconium in pure argon (see Fig. 2,a) and in mixture of argon with oxygen (see Fig. 2,b).

As can be seen from Fig. 2,a, the CVCs for magnetrons with Al and Zr targets are significantly different for pure argon, while the influence of each magnetron to other is not observed.

In Fig. 2,b the CVCs for the Al and Zr targets are shown for the mixture of argon and oxygen. As can be seen from the figure, all the CVCs are S-shaped, and consist of the transition region and two saturation regions: the higher one for pure argon, and the lower one appearing in the target passivation mode at sufficiently high flow of oxygen. There is a region with a negative slope for medium flow values of oxygen. One can also see that a hysteresis is observed in the transition region. The width of the hysteretic loop increases at the reactive gas flow increase. The S-shaped curve can be passed completely for small gas flow values, but above a certain threshold value the slope of the S-curve becomes greater than the slope of the load curve of our power supply and abrupt transition happens from the passivation regime to the "metallic" mode. With further reactive gas flow increase, the exit current from the passivation appears too high, so the power of the power supply is insufficient.

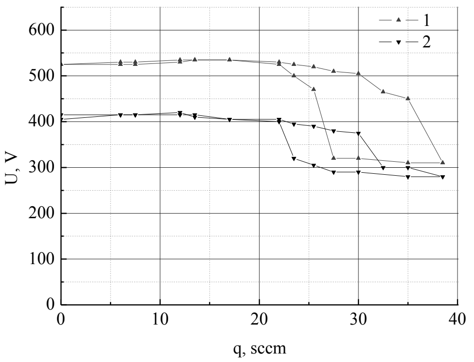
When two magnetron operate (see lines 3, 4 in the Fig. 2,b) then the significant influence of one magnetron on the other is observed. The hysteresis of CVC of each magnetron shifts to lower values of the discharge current.

The Fig. 3 demonstrates the hysteretic of magnetron voltage versus the oxygen flow rate. Fig. 3,a shows that the passivation of the two magnetron targets does not occur simultaneously. When one magnetron enters the passivation mode then the voltage on the second magnetron decreases stepwise, that is caused by sharp increase of oxygen partial pressure in the chamber. Further increase of the oxygen flow induces the second magnetron transition to the passivation mode. Exit from the passivation mode occurs in reverse order.

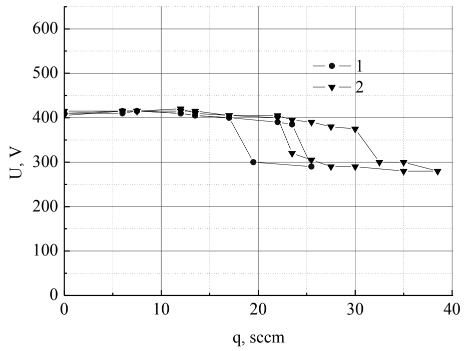
If one chooses the power of the magnetron discharge so that the transition to the passivation mode of both magnetrons occur for similar flow of reactive gas, then the picture is somewhat different one. When the first magnetron reaches the critical oxygen flow, then it switches to the passivation mode thus releasing some additional amount of oxygen, that is sufficient for the second magnetron jump to passivation mode. Thus, both magnetrons switch into the passivation mode (and vice versa) simultaneously. One can also observe from the Fig. 3,b the shift of the hysteresis region to higher values of oxygen flow rate when two magnetrons operate simultaneously.

The obtained results allow choosing the “process window” for the synthesis of oxide coatings of Al, Zr and their combinations. The experiment shows that successful synthesis of stoichiometric oxide film is possible only within a narrow “window” on the oxygen flow rate axis. At lower rates only non-stoichiometric nontrasparent film can be obtained while the oxygen flow rate increase above the “window” causes arcing at the magnetron target leading to contamination of the grown film by metallic droplets. Adjusting the position of the plasma source in relation to the magnetrons one can achieve the situation when the “process window” is shifted above the hysteresis by the voltage axis so that the coating process takes place in the “metallic mode” far away from the area of passivation and microarcs.

For Al and Zr targets, the oxide coating should be deposited in the "metallic mode", i.e. when the target is far from passivation. This is necessary to avoid microarcs and, as a consequence, the droplets. These conditions are satisfied at the upper part of the lines higher than the hysteresis.



a



b

Fig. 3. Dependencies of magnetron discharge voltage on the oxygen flow rate: a – Zr (line 1) and Al (line 2) targets; b – single (line 2) and double (line 1) magnetron operation with Al target. Argon pressure $P = 1\times10^{-3}$ Torr

The obtained results allow us to choose the "process window" for the synthesis of oxide coatings of Al, Zr and their combinations.

CONCLUSIONS

Thus, the experimental research of the current-voltage characteristics of the double-magnetron discharge in noble (Ar) and reactive (O) gases for different target materials (Al, Zr) and their interdependence is reported in the present paper. It is shown that in pure Ar any mutual influence of two magnetrons is absent. On the contrary, the interference between two magnetrons causes the hysteretic region shift in CVC towards lower currents in the case of oxygen usage as a reactive gas. The similar shift in the magnetron discharge voltage dependence on the oxygen flow rate is observed towards higher values of the flow rate. Basing on the research results it is found that it is most expedient to deposit the oxide coatings at the top branch of the CVC, i.e. in "metallic mode".

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КЛАСТЕРНАЯ УСТАНОВКА С ДВУМЯ МАГНЕТРОНАМИ ДЛЯ СИНТЕЗА МИКРО- И НАНОСТРУКТУРНЫХ ПОКРЫТИЙ

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Представлены результаты исследования технологических режимов реактивного магнетронного напыления в кластерной установке с двумя плоскими магнетронами и источником плазмы. Представлены ВАХ магнетронов, а также зависимости тока и напряжения магнетрона и давления в камере от потока реактивного газа с акцентом на особенностях совместной работы двух магнетронов с мишенями из различных материалов. На основании измеренных характеристик определено оптимальное "технологическое окно".

КЛАСТЕРНА УСТАНОВКА З ДВОМА МАГНЕТРОНАМИ ДЛЯ СИНТЕЗУ МІКРО- ТА НАНОСТРУКТУРНИХ ПОКРИТТІВ

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Представлені результати дослідження технологічних режимів реактивного магнетронного напилювання в кластерній установці з двома плоскими магнетронами і джерелом плазми. Представлено ВАХ магнетронів, а також залежності струму і напруги магнетрона та тиску в камері від потоку реактивного газу з акцентом на особливостях спільної роботи двох магнетронів з мішенями з різних матеріалів. На базі виміряних характеристик визначено оптимальне "технологічне вікно".