TECHNOLOGICAL APROBATION OF INTEGRAL CLUSTER SET-UP FOR COMPLEX COMPOUND COMPOSITES SYNTESIS

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In the present paper the results of technological approbation of the integral cluster set-up for synthesis of various types of high-quality coatings such as Al_2O_3, TiO_2, ZrO_2, AlN, TiN, and others with coating thickness up to 10 mkm are presented. Current-voltage characteristics of magnetron discharge in argon mixtures with oxygen and nitrogen for various gas pressures and for various target materials have been measured.

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INTRODUCTION

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

The research results of 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 created the experimental multifunctional cluster ion-plasma system with parameters corresponding 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 TiN, AlN, TiO_2, Al_2O_3, ZrO_2 and their combinations.

The tasks of this work were the measurement of current-voltage characteristics of magnetron discharge in argon mixtures with oxygen and nitrogen for various gas pressures and for various target materials and analysis of the dependences of discharge parameters on target material, pressure and composition of working gas.

1. EXPERIMENTAL SETUP

The cluster set-up is schematically shown in the fig.1. The system consists of the low-pressure magnetron 2 located on the butt end of chamber, the RF inductive source of plasma and activated particles of reactive gas 3 located inside the chamber, the ion source 8 located on lateral flange of the chamber. The relative location of these components has been 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 gase.

In the system a planar magnetron with permanent magnets was used (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.

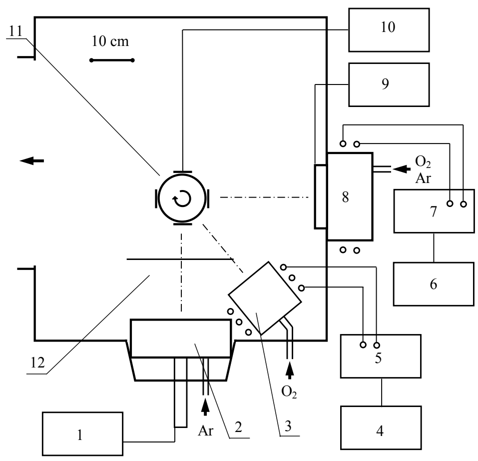

Fig. 1. Scheme of the cluster set-up for complex composite compounds synthesis. 1 – DC magnetron power supply, 2 – magnetron, 3 – RF ICP source, 4, 6 – RF generator, 5, 7 – RF matchbox, 8 – ion source, 9 – DC power supply, 10 – pulsed power supply for samples polarization, 11 – samples rotation system, 12 – shutter

2. EXPERIMENTAL RESULTS

The oxide Al_2O_3 and ZrO_2 coating deposition was performed in high vacuum pumping system with the base pressure about 10^{-5} mBar. There was the problem of target oxidation during deposition process. At 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. Also, such deposition conditions allow to avoid micro-arcs and micro-drops formation increasing the corrosion resistance properties.

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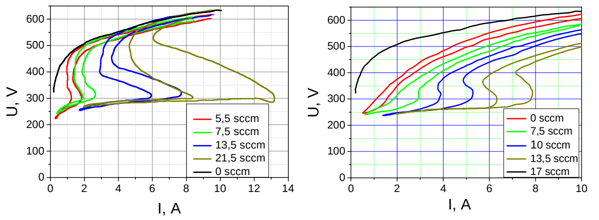

Fig. 2. CVC of magnetron discharge for different flows of oxygen (left) and nitrogen (right). Argon pressure $p = 1.5 \times 10^{-3}$ Torr, the target material: aluminum

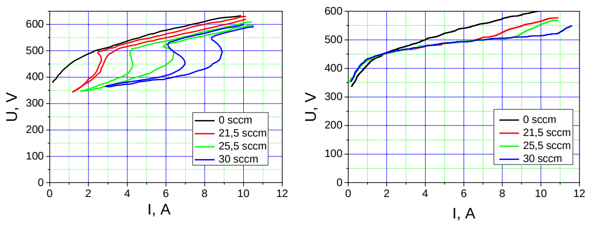

Fig. 3. CVC of magnetron discharge for different flows of oxygen (left) and nitrogen (right). Argon pressure $p = 1 \times 10^{-3}$ Torr, the target material: zirconium

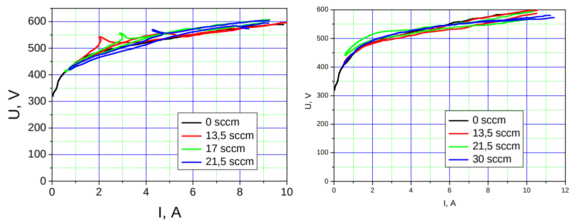

Fig. 4. CVC of magnetron discharge for different flows of oxygen (left) and nitrogen (right). Argon pressure $p = 1 \times 10^{-3}$ Torr, the target material: titanium

The optimum conditions were realized for the upper part of VAC curves of magnetron discharge in argon with oxygen both for aluminum and zirconium target materials

Figs. 2-4 presents the current-voltage characteristic (CVC) of the magnetron with targets of aluminum, zirconium and titanium in a mixture of argon with oxygen or nitrogen at various reactive gas flows. In Fig. 2 the current-voltage characteristics for the aluminum target are shown. As can be seen from the figure, the CVC is S-shaped, and consists of the transition region and two saturation regions: the higher for pure argon, and the lower appearing in the target passivation mode at sufficiently high flow of oxygen. For medium flow values of oxygen there is a region with a negative slope. One can also see that in the transition region a hysteresis effect is observed. At the reactive gas flow increase the width of the hysteretic loop increases. For small gas flow values the S-shaped curve can be passed completely, 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 nitrogen is used with the aluminum target (Figure 2), we observe a pronounced hysteresis in the form of S-shaped curve similar to the oxygen case.

Fig. 3 shows the magnetron current-voltage characteristics for zirconium target. With the oxygen used as a reactive gas the same effect is observed, but the S-shaped curve is less pronounced on the branch with negative slope, the effect of hysteresis is also pesent. As in the case of the aluminum target, at the oxygen flow increase the curves are shifted to the right. When the zirconium target is used with nitrogen (Fig. 3), there is almost no hysteresis.

For aluminum and zirconium targets the oxide coating deposition process must be done 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 curves higher than the hysteresis.

Fig. 4 shows the current-voltage characteristics for titanium target, which looks differently. As we can see, there is also the phenomenon of hysteresis, but the loop is N-shaped and crossed herself. In contrast to the CVC for aluminum, CVC for titanium and zirconium cross the curve for pure nitrogen: the high power curves for nitrogen are lower, and the small power ones are higher than the curve for pure nitrogen. As can be seen from the figure, the higher the nitrogen flow, the higher the intersection point lies.

The obtained results allow us to choose the "process window" for the synthesis of oxide and nitride coatings of aluminum, zirconium and titanium.

Fig. 5 shows the surface topography of ZrO_2 obtained using an atomic force microscope. Table lists some of the mechanical and tribological properties of the deposited coatings.

CONCLUSIONS

Thus, in the present paper the experimental research of the current-voltage characteristics of the magnetron discharge in inert (argon) and reactive gases (oxygen, nitrogen) for different target materials (aluminum, zirconium, titanium) is reported. It is found that for pure argon for all target materials and for all investigated pressures the same shape and behavior of the CVC is observed. With oxygen filling the pronounced hysteresis effect is observed for all the targets: for aluminum and zirconium there is a S-haped curve while for titanium target the curve is N-shaped, hat is consistent with the similar characteristic

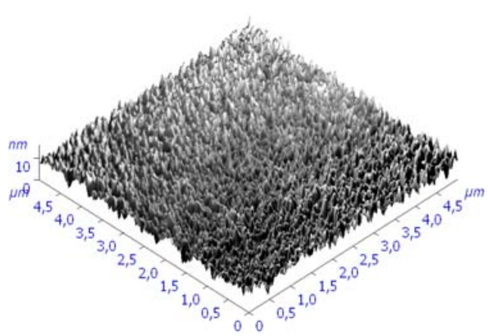

Fig. 5. The surface topography of ZrO_2 obtained by AFM

Mechanical and tribological characteristics of oxide coatings Al_2O_3 and ZrO_2

Material/ Coating type	Mechanical parameters (average results of 10 tests)			
	Hardness Hv	Hardness H [Mpa]	Young Modulus [Gpa]	Adhesion [N]
Zr/ZrO_2	755.5	7831.5	167.7	28.5
Zr/ Al_2O_3	782.0	8115.2	184.4	27.1
Ti/ZrO_2	767.5	8072.4	172.3	38.4
Ti/ Al_2O_3	953.6	8289.9	197.0	40.3

in [5]. For nitrogen filling the hysteresis effect is present in the CVC for aluminum (similar to that for oxygen) and titanium. In the CVC for zirconium no hysteresis effect is observed.

Basing on the research results it has been found that the deposition of the oxide and nitride coatings is most expedient to perform at the top branch of the CVC, i.e. in "metallic mode".

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ТЕХНОЛОГИЧЕСКАЯ АПРОБАЦИЯ ИНТЕГРАЛЬНОЙ КЛАСТЕРНОЙ УСТАНОВКИ ДЛЯ СИНТЕЗА СЛОЖНОКОМПОЗИЦИОННЫХ СТРУКТУР

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Представлены результаты технологической апробации интегральной кластерной установки для синтеза высококачественных покрытий типа Al_2O_3, TiO_2, ZrO_2, AlN, TiN и других, толщиной до 10 мкм. Были измерены вольт-амперные характеристики магнетронного разряда в смесях аргона с кислородом и азотом, при различных давлениях рабочего газа и разных материалах мишени.

ТЕХНОЛОГІЧНА АПРОБАЦІЯ ІНТЕГРАЛЬНОЇ КЛАСТЕРНОЇ УСТАНОВКИ ДЛЯ СИНТЕЗУ СКЛАДНОКОМПОЗИЦІЙНИХ СТРУКТУР

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Представлено результати технологічної апробації інтегральної кластерної установки для синтезу високоякісних покриттів типу Al_2O_3, TiO_2, ZrO_2, AlN, TiN та інших, товщиною до 10 мкм. Було виміряно вольт-амперні характеристики магнетронного розряду в сумішах аргону з киснем та азотом, при різних тисках робочого газу та різних матеріалах мішені.