INTEGRAL CLUSTER SET-UP FOR COMPLEX COMPOUND COMPOSITES SYNTESIS

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At present study the result of development and investigations of cluster technological set-up for synthesis of complex compound composites is demonstrated. The present set-up consist of complimentary DC-magnetron system, RF-inductive plasma source and ion source. The 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 nanocomposites. Various types of high-quality coatings such as Al_2O_3, AlN, TiO_2, TiN, ZrO_2 and others with thickness up to 10 mkm have been synthesized using the described set-up.

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1. INTRODUCTION

The planar DC magnetrons are widely used for deposition of metallic coatings [1, 2]. At the present time the synthesis of dielectric films such as TiO_2, Al_2O_3, Ta_2O_5 etc. encounter significant problems at simple reason that these compaunds do not conduct the direct current of charged particles. It results in the magnetron target passivation, intence arcing, "disappearind anode" phenomenon and various discharge instabilities. All of this considerably narrows the technological "window" for synthesis of high-quality coatings [3-5].

To solve these problems the technological module was developed for the reactive synthesis of complex-composite coatings such as oxides and oxynitrides on the base of DC magnetron and RF inductive plasma source [6]. The research results of the different module components were published previously:

- the research of the low-pressure DC magnetron [7];
- the research of arcing processes at the magnetron target in the oxygen atmosphere [8, 9];
- the research of the target passivation [10];
- the research of the RF inductive plasma source [11].

The research of the technological module operation was also conducted in technology of high-quality Al_2O_3 coatings synthesis [12].

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 nanocomposite) coatings and structures based on TiN, AlN, TiO_2, Al_2O_3, ZrO_2 and their combinations.

The basic novelty of the present work is the combination in one cluster vacuum-plasma system of few compatible sources of flows of metals, ions, chemically-active particles for the sequential or simultaneous processing of micro- and nano-structures.

2. 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 gas.

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 is made of aluminum, zirconium and titan. 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.

The RF inductive plasma source 3 serves as plasma activator of the reactive gas, and also produces a stream of slow ions and electrons. It is applied for the surface clearing of workpieces and is compatible with the magnetron for deposition of metal oxides and nitrides. The plasma source is placed inside the vacuum chamber that allows to choose the optimum relation between the distances from the magnetron to samples and from the

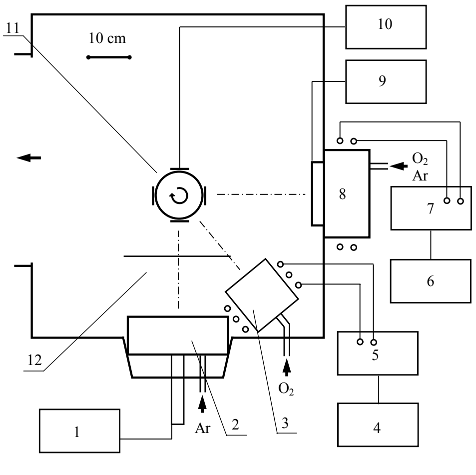

Fig. 1. Scheme of the cluster set-up for complex composite compounds synthesis. 1 – DC magnetron power supply; 2 – magnetron; 3 – RF ICP source; 4; 6 – RF generator; 5, 7 – RF matchbox; 8 – ion source; 9 – DC power supply; 10 – pulsed power supply for samples polarization; 11 – samples rotation system;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

plasma source to samples. Plasma in such source is concentrated in the discharge chamber made of a ceramic tube (Fig. 3). At the source exit the perforated metal screen is erected which restricts the plasma and provides a pressure drop between the source volume and the technological chamber.

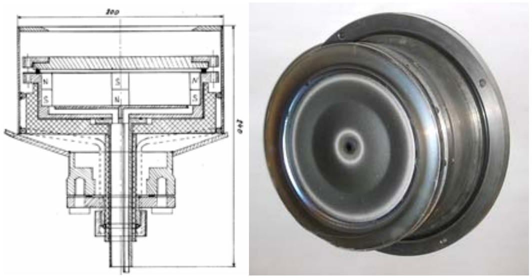

Fig. 2. Low-pressure magnetron

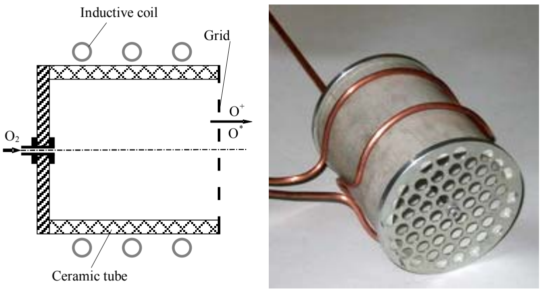

Fig. 3. RF ICP source

The RF inductive plasma source is supplied with the RF power up to 1 kW (frequency 13.56 MHz) by the RF generator 4 which is connected to the inductive coil by trough the RF matchbox 5.

The ion source 8 produces the 50…500 eV ion beam directed to the processed samples and can be applied as standalone device for etching of the samples, the ion polishing, and cleaning the surface before the coating process as well as simultaneously with the magnetron discharge for synthesis of coatings with different unique properties. The construction of the ion source illustrated by the Fig. 4.

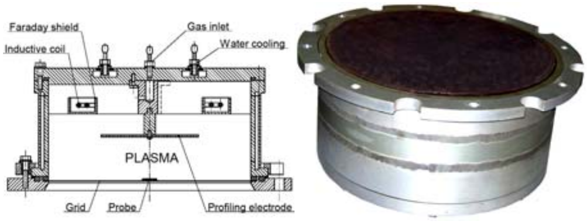

Fig. 4. Single-grid RF ion source

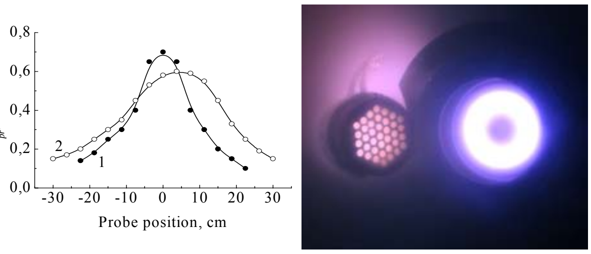

Fig. 5. Radial distributions of ion current density to the substrate holder for magnetron plasma (1) and ICP (2)

Using the pulsed power supply 10 for the workpieces polarization it is possible to apply a constant voltage or impulses of different duty cycle to the rotated substrate holder 11.

3. EXPERIMENTAL RESULTS

The basic idea of the plasma activated reactive synthesis of oxide coatings consists in the separation of two processes: metal target sputtering using a direct current magnetron in inert gas and activation of the reactive gas by means of the additional plasma source based on the RF inductive discharge. The important condition of the technology is to deliver the activated reactive gas as close as possible to the processed surface increasing the effective partial pressure of the reactive gas in the synthesis region. In this case the useful range of parameters is much wider due to keeping the target in metallic mode.

In Fig. 5 the radial distribution of the ion current density to the substrate holder is presented separately for the magnetron plasma and RF inductive plasma. At the working pressure the streams of ions and species are distributed approximately at cosine law.

This technology was initially developed for the Al_2O_3 coatings synthesis [6, 12], but to the moment it has been adopted to synthesis of other dielectric materials such as ZrO_2.

For the synthesized coatings the researches of adhesion, properties of elasticity and plasticity, corrosion durability have been carried out.

The most interesting property of the dielectric coatings like Al_2O_3, ZrO_2 is their unique corrosion resistance. The corrosion resistance of single-layer and multilayer structures Al_2O_3, TiN/Al_2O_3, Al/Al_2O_3 has been measured using the electrochemical method in relation to corrosion resistance of SW7M steel (Fig. 6). One can see from the Fig.6 that the combination TiN/Al_2O_3 has the highest corrosion resistance.

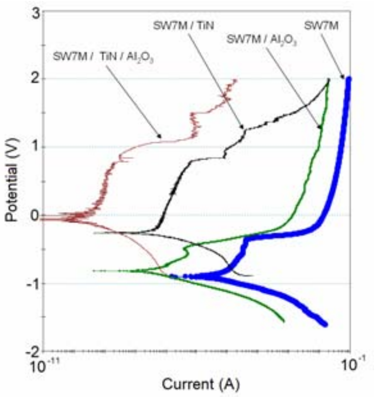

Fig. 6. Anode polarization curve, obtained by potentiodynamic method for SW7M steel sample without a coating and with TiN and Al_2O_3 coatings

4. CONCLUSIONS

The multifunctional cluster ion-plasma technological system for synthesis and processing of complex composite (including nanocomposite) multilayer coatings and structures in particular TiN, AlN, TiO_2, Al_2O_3, and their combinations has been created. The parameters of the system correspond the demands of industrial operation:

Magnetron power – to 10 kW;
Size of processed surface – to 500 mm;
Deposition rate – to 10 μm/hr;
Process cycle – about 2 hours.

The system consists of the following complementary plasma modules:

1. The low pressure unbalanced magnetron ;
2. The RF inductive source of plasma and activated reactive gas;
3. The ion source;
4. DC/pulsed samples polarization system .

The basic novelty of the present work is the combination in one cluster vacuum-plasma system of few compatible sources of flows of metals, ions, chemically-active particles for the sequential or simultaneous processing of micro- and nanostructures.

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ИНТЕГРАЛЬНАЯ КЛАСТЕРНАЯ УСТАНОВКА ДЛЯ СИНТЕЗА СЛОЖНОКОМПОЗИТНЫХ СТРУКТУР

С.Д. Яковин, С.В. Дудин, А.В. Зыков, В.И. Фареник

Представлены результаты разработки и исследования технологической установки для синтеза сложнокомпозитных соединений на поверхности. Установка состоит из комплементарных модулей магнетрона постоянного тока, ВЧ-индукционного источника плазмы и источника ионов. Система позволяет независимо формировать потоки металлических атомов, химически активных частиц, ионов и синтезировать тонкие сложнокомпозитные пленки, включая нанокомпозиты. На представленной установке были получены различные типы высококачественных покрытий, таких как Al_2O_3, AlN, TiO_2, TiN, ZrO_2 и др., толщиной до 10 мкм.

ІНТЕГРАЛЬНА КЛАСТЕРНА УСТАНОВКА ДЛЯ СИНТЕЗУ СКЛАДНОКОМПОЗИТНИХ СТРУКТУР

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Представлено результати розробки і дослідження технологічної установки для синтезу складнокомпозитних сполук на поверхні. Установка складається з комплементарних модулів магнетрона постійного струму, ВЧ-індукційного джерела плазми і джерела іонів. Система дозволяє незалежно формувати потоки металевих атомів, хімічно активних часток, іонів і синтезувати тонкі складнокомпозитні плівки, включаючи нанокомпозити. На цій установці було отримано різні типи високоякісних покриттів, таких як Al_2O_3, AlN, TiO_2, TiN, ZrO_2 та ін., товщиною до 10 мкм.