SYNTHESIS OF THIN-FILM TA_2O_5 COATINGS BY REACTIVE MAGNETRON SPUTTERING

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The investigation results of optimal conditions for synthesis of thin-film tantalum oxide dielectric coatings using the cluster multipurpose setup are presented. The set-up consist of DC magnetron, ICP source, and medium-energy ion source. Tantalum oxide was deposited by reactive magnetron sputtering using DC magnetron in atmosphere of argon and oxygen. The oxygen flow was activated by passing trough the ICP source. The described equipment allows independent control of the flows of metal atoms, of reactive particles, and of ions of rare and reactive gas. The current-voltage characteristics of the magnetron discharge were measured as well as teir dependencies on argon pressure and oxygen flow.

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INTRODUCTION

Tantalum, the transition metal, possesses unique physical and chemical properties. So, yielding up only to tungsten by melting point (T_m= 3017°C), tantalum has outstanding chemical durability, considerably excelling on this parameter such metal, as gold. A tantalum is a good conductor and unique metal which is not torn away by human tissue [1, 2].

A higher oxide of tantalum Ta_2O_5 is an excellent dielectric (ε = 28…32), has high mechanical characteristics, is chemically and biologically inert. After the special treatment it acquires electret properties [3]. Ion-plasma technologies of synthesis of Ta_2O_5 stoichiometric coatings provide the control of electret properties in the deposition process. It is succeeded to get coatings with the effective surface charge density more than $1{\cdot}10^{-4}$ K/m^2 keeping practically unchanged properties during long time [4].

Getting together with an implant in the human organism the electretic film affects locally the damaged organ contributing to its treatment in optimum biological terms. This process has a natural effect consisting of that external short-range electric field of certain value and sign, operating at cellular level, is the catalyst of appearance of new healthy formations in living texture. Therefore a new rise in area of prospective biocoatings is associated with the use of new dielectric and electret coatins of tantalum pentoxide [5, 6].

In previous study the results of elaboration and investigations of cluster technological setup for synthesis of complex compound composites were demonstrated [7]. The presented set-up consists of complimentary DC-magnetron system, RF-inductive plasma source and ion source. The system allows to control independently 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 [8];
- the research of arcing processes at the magnetron target in the oxygen atmosphere [9];
- the research of the target passivation [10];
- the research of the ICP source [11].

On the base of this module we created 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.

In the present paper the results of technological regimes investigation of thin-film Ta_2O_5 deposition using reactive magnetron sputtering are presented in comparison with analogous results for Al_2O_3. The depositions performed in the cluster set-up comprising planar magnetron, plasma source and medium energy ion source. On the basis of the measured characteristics the technological "window" is determined and some properties of deposited Ta_2O_5 coating are investigated.

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, and the ion source 6 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 is used. The magnetron power supply allows to bias the magnetron target at up to 1 kV negative potential with the discharge current up to 20 A, maximum power of the supply is 6 kW. The magnetron target of 170 mm diameter is made of tantalum. 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 to 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 may operate simultaneously with the magnetron and was applied for deposition of metal oxides. 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 plasma source to samples.

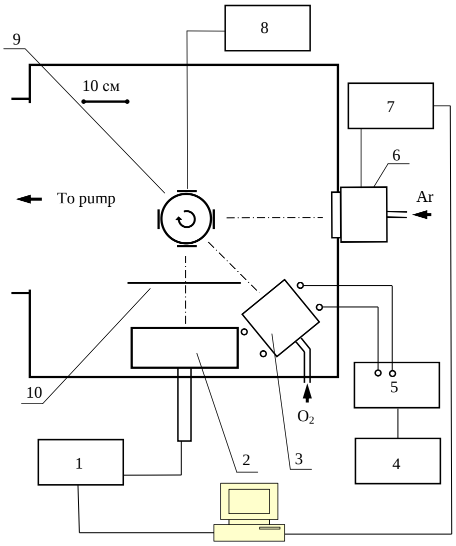


Fig. 1. Scheme of the cluster set-up for complex composite compounds synthesis. 1 – DC magnetron power supply; 2 – magnetron; 3 – RF ICP source; 4 – RF generator; 5 – RF matchbox; 6 – ion source; 7 – probe; 8 – DC power supply power supply for samples polarization; 9 – samples rotation system; 10 – shutter

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

The ion source 6 "Radical M" [12] produces the ion beam with mean energy of 0.5…1.5 keV directed to the processed samples and can be applied as standalone device for etching of the samples and cleaning the surface before the coating process as well as simultaneously with the magnetron discharge for synthesis of coatings with different unique properties.

Using the pulsed power supply 8 it is possible to apply a constant or pulsed voltage of different duty cycles to the rotated substrate holder 9 for the samples polarization.

2. EXPERIMENTAL RESULTS

In order to determine optimal conditions for synthesis of tantalum oxide dielectric coatings the current-voltage characteristics (CVC) of the magnetron discharge, and dependencies of the current and the voltage on the argon pressure and oxygen flow were measured.

Figs. 2, 3 presents the current-voltage characteristic of the magnetron with tantalum target in argon and in a mixture of argon with oxygen at various oxygen flows. As shown in Fig. 2 CVC in argon is weakly dependent on the argon pressure.

Fig. 3 shows the current-voltage characteristics for tantalum target for different oxygen flow rate. As it is seen from the figure the magnetron current-voltage trace in the argon-oxygen mixture is N-shaped and hysteretic. In the whole discharge current range the curves lay higher then the characteristics for pure argon. The right side of the trace represents the "metal" target mode while the left side corresponds to passivated target surface. The region of stoichiometric Ta_2O_5 coatings deposition is in between the dashed lines.

The dependencies of magnetron discharge voltage and current vs. oxygen flow rate (Fig. 4) also demonstrates hysteresis effect. As it is seen from the figure in the "passivation" mode the magnetron voltage increases by 20 % while the magnetron current show a threefold drop.

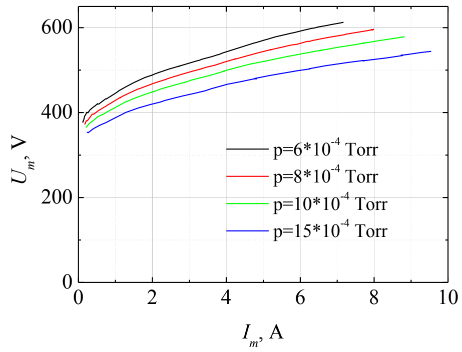


Fig. 2. CVC of magnetron discharge for different argon pressure. The target material: tantalum

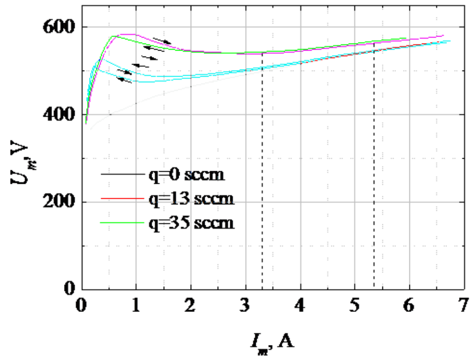


Fig. 3. CVC of magnetron discharge for different flows of oxygen. Argon pressure p = 8·10^{-4} Torr, the target material: tantalum

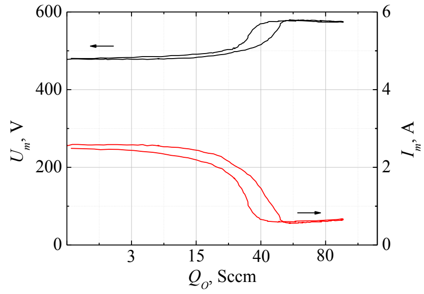


Fig. 4. Voltage and current curves of magnetron discharge vs oxygen flow. Argon pressure p = 8·10^{-4} Torr, the target material: tantalum

In the Fig. 5 CVC for tantalum and aluminum targets at pure argon and Ar/O_2 mixture are presented for comparison. As can be seen from the Fig. 5, the CVC for Al 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. The CVC for tantalum in Ar/O_2 mixture is N-shaped and the discharge voltage is higher than the same one for pure argon.

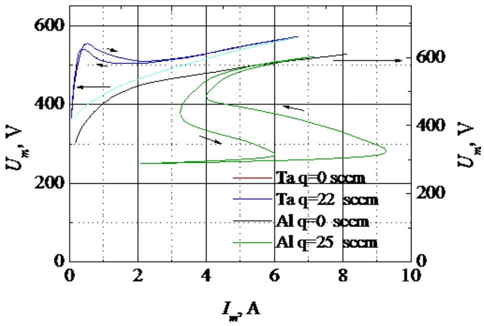


Fig. 5. CVC of magnetron discharge for tantalum and aluminum targets. Argon pressure p = 8·10^{-4} Torr

3. TECHNOLOGICAL REGIME

The obtained results allow us to choose the "process window" for the synthesis of oxide tantalum coatings. As in the case of alumina coatings it is advantageous to carry out the Ta_2O_5 synthesis in "metallic" mode when the target is far from the passivation. Unlike the alumina case the tantalum target does not demonstrate the dramatic drop in the magnetron voltage. However, as can be seen from the Fig. 4, the discharge power and consequently the deposition rate drops significantly with the oxygen flow rate growth. So, the limiting factors for the "technological window" are the deposition rate drop at high oxygen flow and non-stoichiometry of the deposited coating from the opposite side (see Fig. 3).

Thus, the sputtering process was conducted in the modes far from the target poisoning and simultaneously providing stoichiometric composition of the deposited film. Also, such deposition conditions allowed us to avoid micro-arcs and micro-drops formation. The optimum conditions were implemented in the right part of the volt-ampere characteristic curves of a magnetron discharge in argon with oxygen.

The main parameters during the technological processes were monitoring by PC and the typical time dependences of these parameters and technological steps for deposition of Ta_2O_5 single-layer and Ta_2O_5/Ta multilayer films are presented in the Figs. 6, 7.

The tantalum pentoxide ceramic coatings deposited by magnetron sputtering process had been investigated in [13, 14]. The coatings were transparent and amorphous and the surface showed no cracks. In [13] the influence of ion bombardment onto the properties of the tantalum oxide film has been investigated while un [14] our coatings are compared with the coatings deposited by electron-beam evaporation.

Fig. 8 shows the surface photos of Ta_2O_5 obtained using transmission electron microscopy (TEM) and atomic force microscopy (AFM).

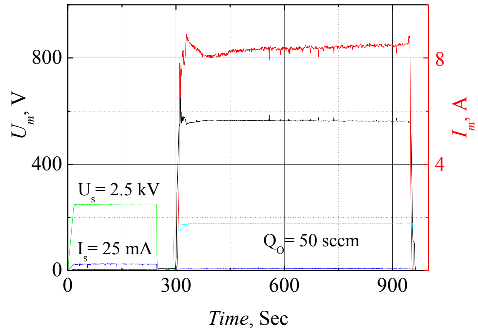


Fig. 6. Single-layer process. 1 – samples cleaning; 2 – film deposition

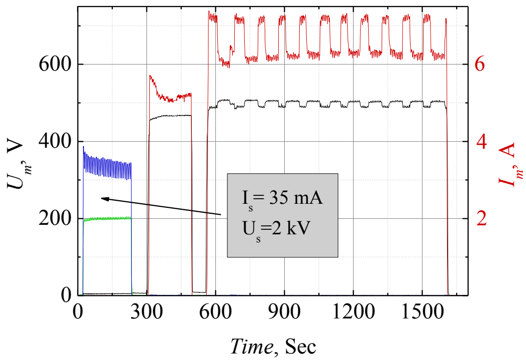
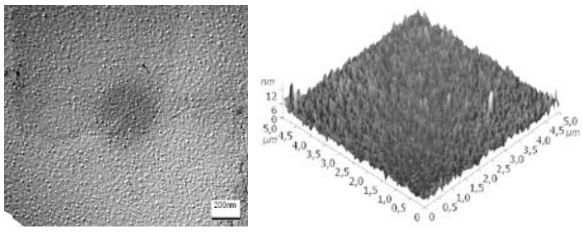


Fig. 7. Multilayer process. 1 − samples cleaning; 2 – target training; 3 – Ta_2O_5/Ta film deposition



a b

Fig. 8. TEM image (a) and AFM surface image (b) of oxide ceramic coating Ta_2O_5

CONCLUSIONS

Thus, in the present paper the experimental results of the current-voltage characteristics research of the magnetron discharge in inert (argon) and reactive (oxygen) gases are presented as well as the dependences of the magnetron current and voltage on the reactive gas flow in the case of tantalum target.

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

On the basis of the measured characteristics the technological "window" is defined. Single-layer and multilayer tantalum oxide coating were produced and investigated.

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СИНТЕЗ ТОНКИХ ПЛЁНОК Ta_2O_5 МЕТОДОМ РЕАКТИВНОГО МАГНЕТРОННОГО РАСПЫЛЕНИЯ

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Представлены результаты исследований для нахождения оптимальных условий синтеза диэлектрических тонких плёнок оксида тантала в кластерной многофункциональной установке. В установку входят: магнетрон постоянного тока, индукционный источник плазмы и источник ионов средних энергий. Оксид тантала наносили реактивным магнетронным распылением с помощью магнетрона постоянного тока в смеси аргона и кислорода. Для активации поток кислорода пропускали через индукционный источник плазмы. Данное оборудование позволяет независимо контролировать потоки атомов металла, активированных частиц и ионов инертного или реактивного газов. Были измерены вольт-амперные характеристики магнетронного разряда, зависимости тока и напряжения разряда от давления аргона и потока кислорода. Покрытия Ta_2O_5 были синтезированы при различных условиях.

СИНТЕЗ ТОНКИХ ПЛІВОК Ta_2O_5 МЕТОДОМ РЕАКТИВНОГО МАГНЕТРОННОГО РОЗПИЛЮВАННЯ

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Представлено результати досліджень для знаходження оптимальних умов синтезу діелектричних тонких плівок оксиду танталу в кластерній багатофункціональній установці. В установку входять: магнетрон постійного струму, індукційне джерело плазми і джерело іонів середніх енергій. Оксид танталу наносили реактивним магнетронним розпиленням за допомогою магнетрона постійного струму в суміші аргону і кисню. Для активації потік кисню пропускали через індукційне джерело плазми. Дане обладнання дозволяє незалежно контролювати потоки атомів металу, активованих частинок та іонів інертного або реактивного газів. Були виміряні вольт-амперні характеристики магнетронного розряду, залежності струму та напруги розряду від тиску аргону і потоку кисню. Покриття Ta_2O_5 були синтезовані при різних умовах.