INVESTIGATION OF INTERACTION BETWEEN ION-BEAM PLASMA AND PROCESSED SURFACE DURING THE SYNTHESIS OF TANTALUM DIBORIDE AND PENTAOXIDE

S. Yakovin, A. Zykov, S. Dudin, A. Dakhov, N. Yefymenko

V.N. Karazin Kharkiv National Universty, Kharkiv, Ukraine

E-mail: stanislav.yakovin@karazin.ua

The paper is devoted to investigation of spatial distributions of ion current density to a sample in technological set-up with magnetron sputtering system and ICP source. The dependence of the ion flux towards the processed surface on the parameters of the deposition process was measured. The following parameters were varied: magnetron discharge power, gas type and pressure, target-sample distance, inductive discharge power, and bias potential applied to the samples. The effect of nonequilibrium heating of the sample surface due to relaxation of kinetic energy of ions, atoms and electrons, as well as energy of exothermic chemical reactions at synthesis of Ta_2O_5 and TaB_2 films is discussed. The influence of sample shape on the ion bombardment is also investigated.

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INTRODUCTION

Last years low-pressure magnetron sputtering with ion-beam or plasma assistance is actively investigated for application in technologies of thin-film coatings deposition [1-5]. The flow of high energy ions allows cavities elimination in the growing film, creation of additional activation centers, stimulation of surface chemical reactions. In the last decade, magnetron sputtering systems have become one of the main methods for deposition of nanocomposite functional coatings, while the additional ion bombardment of the substrate is carried out by variety of methods and allows control of internal stresses, microstructure and macro properties of deposited films. For example, it was shown in [2] that the argon ions bombardment during tantalum pentoxide coating deposition process reduces the adhesive and proliferative potential of bone marrow cells. Ion bombardment can be used for the structure control of deposited coatings. The evolution of XRD pattern of TaB_2 coating deposited by magnetron sputtering with different sample bias shown in [5] exhibits significant impact of ion bombardment on the coating crystallinity.

Thus, the ion bombardment of the growing film is of crucial importance for the properties of deposited coatings. Usually, its efficiency is defined by the two factors: ion energy and current density. The ion energy for conductive substrates and coatings is usually controlled by a negative bias applied to the substrate, that provides possibility to control easily the energy in wide range. In contrast, the ion flow density is frequently defined by plasma surrounding the sample and can not be controlled independently from deposition source parameters.

One of the most widely used plasma sources is the Inductively Coupled Plasma (ICP) source. In the case of simultaneous operation of the source and magnetron discharge an interaction between the ICP, the magnetron plasma and the processed surface can play an important role, but it is not studied.

The present paper is devoted to investigation of spatial distributions of ion current density to a sample in technological set-up with magnetron sputtering system and ICP source. The paper presents the studies results of a nonequilibrium plasma parameters in the multipurpose cluster setup [6, 7] comprising a DC magnetron, an ICP source [6, 8], and a medium-energy ion source [9, 10]. The equipment allows independent control of the flows of metal atoms, of reactive particles, and of ions of rare and reactive gas.

The dependence of the ion flux towards the processed surface on the parameters of the deposition process was measured. The process parameters which were varied are the following: magnetron discharge power, gas type and pressure, target-sample distance, inductive discharge power, and bias potential applied to the samples. The influence of sample shape on the ion bombardment is also investigated. The obtained results are useful for investigation of effect of nonequilibrium heating of the sample surface due to relaxation of kinetic energy of ions, atoms and electrons, as well as energy of exothermic chemical reactions at synthesis of Ta_2O_5 and TaB_2 films.

1. EXPERIMENTAL SET-UP

The set-up is schematically shown in Fig. 1. The system consists of the low-pressure magnetron 2 with target diameter of 170 mm and the RF ICP source of activated particles of reactive gas 3 placed inside 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. Power supplies are able to deliver up to 6 kW to the magnetron and up to 1 kW to the plasma source. Residual pressure was about 10^{-3} Pa, while working gas pressure varied in the range $(5…10)\cdot10^{-2}$ Pa. Experiments were conducted using Argon and Oxygen, and there was a possibility to feed the gas immediately to the chamber, or to the plasma source vessel. In the last case a pressure drop occurs between the source and the chamber.

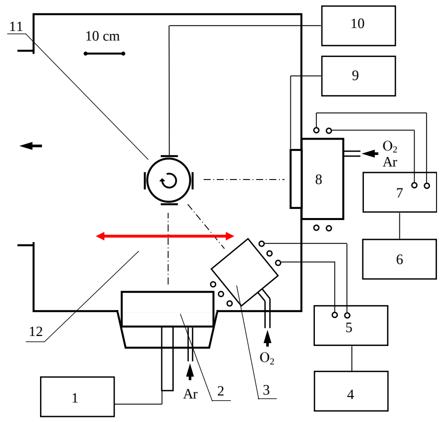

Fig. 1. Scheme of the experimental set-up:
1 – DC magnetron power supply; 2 – magnetron;
3 – RF ICP source; 4, 6 – RF generator;
5, 7 – RF matchbox; 8 – ion source; 9 – DC power supply; 10 – pulsed power supply for samples polarization;
11 – samples rotation system; 12 – probe trajectory

1.1. MEASUREMENT TECHNIQUE OF RADIAL DISTRIBUTIONS OF ION CURRENT

In order to measure spatial distribution of ion current density a flat probe of 5 mm diameter was put into the chamber. The probe mounting allowed to move the probe without vacuum break along the trajectory shown in the Fig. 1. The distance from the probe to the magnetron target was 250 mm. Potentiometer mechanically connected to L-shaped probe shaft allowed to convert rotation angle to voltage. This coordinate signal along with the probe current signal (measured as the voltage drop on a shunt resistor) was measured using an analog-to-digital converter and recorded by a computer.

An important question for our measurements was the probe biasing. Usually, it is enough to apply to the probe a few tens of Volts of negative bias to be in ion saturation region of the current-voltage characteristics of the probe. However, magnetron discharge is known as intense electron source, and electron energies might be quite significant. Thus before the main measurements the influence of the probe bias on the measurement result was investigated.

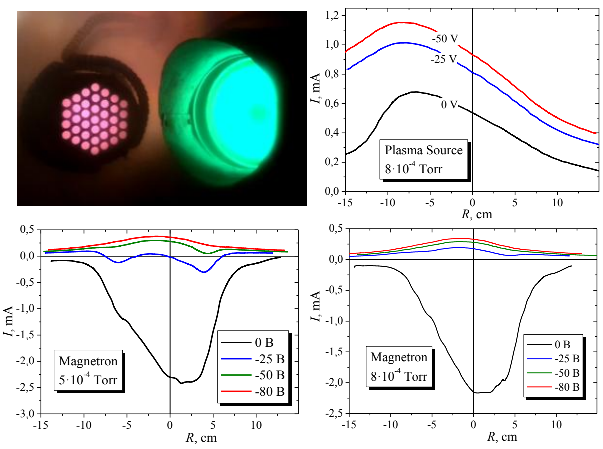

Fig. 2. Photo of relative arrangement of plasma source and magnetron; spatial distributions of probe current at different probe biasing

The spatial distributions of probe current at different probe biasing are shown in Fig. 2 separately for magnetron and plasma source. It can be seen that -25 V bias is enough to measure ion current from ICP, while at least -50 V is necessary to suppress electron flow from the magnetron at the pressure of $8 \cdot 10^{-4}$ Torr. Even more voltage (-80 V) is required at lower pressure of $8 \cdot 10^{-4}$ Torr. All the following measurements were done with the probe bias of -80 V.

2. EXPERIMENTAL RESULTS

Spatial distributions of ion current was studied experimentally for the plasma source and the magnetron separately, while at the final stage simultaneous operation of the both devices was researched. Gas pressure and discharge power appear as variable parameters. Additionally the influence of pressure difference between the plasma source and main chamber was investigated as well as the impact of gas type (Argon, Oxygen) on the ion flow to the processed surface. One more important factor influencing the ion current density to the surface is the surface shape. Depending on the shape ion flow may be focused or defocused changing the current density by few times. The results of investigation of ion current density response to variation of the mentioned factors are presented below.

2.1. PLASMA SOURCE: DEPENDENCE ON DISCHARGE POWER

The experiments was done at Argon pressure in the chamber of $5 \cdot 10^{-4}$ Torr. Radial distributions of the ion current density are shown in Fig. 3 for different RF power input to the ICP source. First of all, one can see significant asymmetry of the distributions. Due to asymmetric placement of the plasma source they are shifted towards its position. Fig. 3 also shows the dependence of maximum ion current density on the RF power. One can expect linear dependence, but at low powers the current has lower derivative on the RF power. In order to understand this behavior of ion current, a series of photos was taken for different RF powers (Fig. 4).

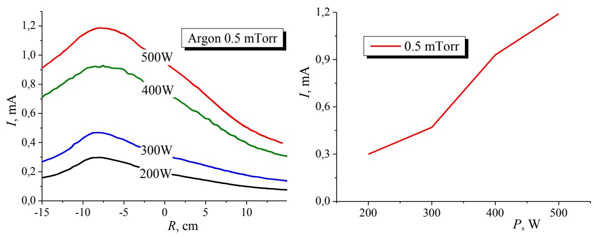

Fig. 3. Radial distributions of ion current density for different RF power input to the ICP source and dependence of maximum ion current density on the RF power

It can be seen from the figure that the glow intensity from the source vessel does not depend on RF power. Moreover, it even drops a bit with the power growth. The excessive power is absorbed by the outer plasma. Its brightness grows linearly with the RF power. Note that the ion bombardment of a sample placed outside the plasma source is more intense in the case of existence of the outer plasma.

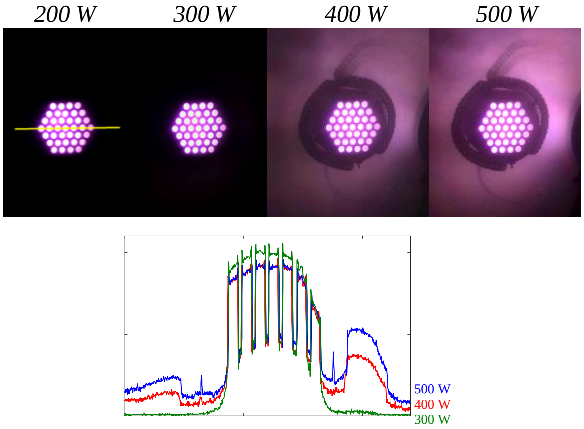

Fig. 4. ICP evolution depending on RF power. Top row: plasma source photos for different RF powers; plot: glow intensity distribution along the marked line

2.2. PLASMA SOURCE: DEPENDENCE ON GAS PRESSURE

Radial profiles of ion current to the probe are shown in Fig. 5 for different Argon and Oxygen pressures. It is important that the gas in this case was fed into the plasma source vessel, so a pressure difference exists between the vessel and the main chamber. The ion current demonstrates a week dependence on Argon pressure (Fig. 5), while significant ion current drop was observed after change Argon to Oxygen at the same pressure. Note that with Argon the plasma was situated both inside and outside the plasma source, while in the Oxygen case the plasma glowed only inside the source vessel. As we seen above the ion bombardment in this case is less pronounced. Another reason for this current drop is higher power loss in the plasma of molecular gases.

Next figure shows the radial distribution of the ion current as well as the photo of plasma glow for the case of Argon feeding to main chamber rather then to the plasma source.

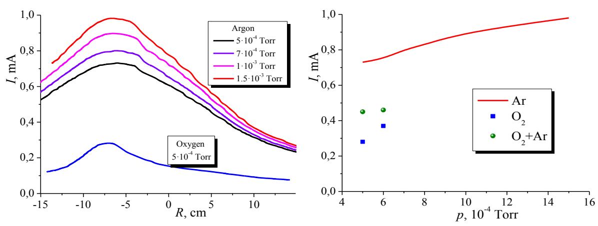

Fig. 5. Radial distributions of ion current density for different Argon and Oxygen pressure; dependence of maximum ion current density on gas pressure

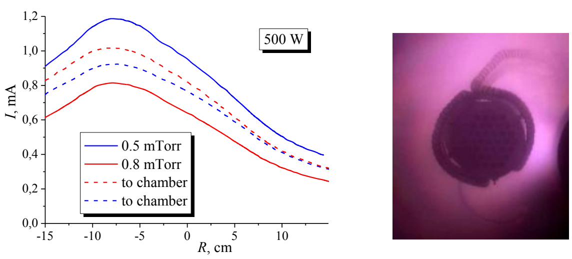

Fig. 6. Radial distributions of ion current density for different Argon pressures with different directions of the gas feeding

One can see that in this case a regime is possible without plasma inside the plasma source. The possibility for plasma to exist in the tree modes (inside, outside, and both) causes changes in the ion current dependence on the gas pressure seen in Figs. 6 and 3. The "plasma in" mode is preferable for reactive gas activation fed trough the ICP source. In this case the RF power is forwarder for molecules dissociation and excitation, while the "plasma out" mode is more suitable for ion bombardment of the processed sample.

2.3. DEPENDENCE ON THE SAMPLE SHAPE

It should be mentioned that the ion current to the plane probe depends not only on the power deposited to the plasma source, but also on plasma boundary conditions. Fig. 7 shows that the ion current to the probe placed in front of the plasma source is approximately 2 times higher than the current to the same probe at the same point and at the same conditions, but in the presence of the substrate holder behind the probe.

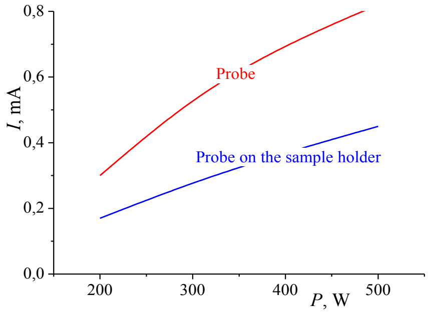

Fig. 7. Comparison of ion currents to the probe with or without the sample holder at the same conditions

2.4. JOINT OPERATION OF MAGNETRON AND PLASMA SOURCE

Fig. 8 shows in comparison the radial distributions of ion current density from plasma source and magnetron for separate and joint operation. It is obvious that ion current from the plasma source is always greater than the current from magnetron plasma, and in the first case the distribution peak is shifted towards the plasma source. One can expect the ion current at joint operation of magnetron and plasma source to be the sum of the currents from the devices. However, measured summary ion current in peripheral regions is even lower then one of the summands. The summary current distribution is noticeably narrower then the one for plasma source.

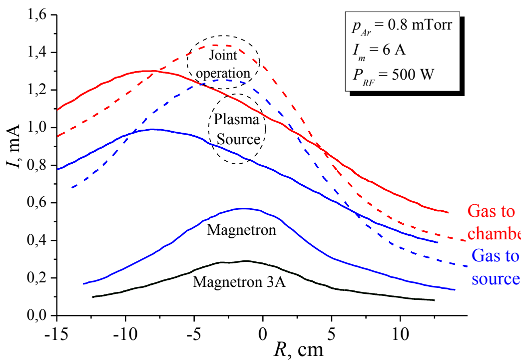

Fig. 8. Radial distributions of ion current density from plasma source and magnetron

CONCLUSIONS

Thus, in the present paper the experimental research of spatial distributions of ion current to a sample in technological set-up with magnetron sputtering system and ICP source is reported. Varying the process parameters (magnetron power, gas type and pressure, ICP power, and sample bias) it has been found that:

– the discharge in the ICP source can operate in 3 modes with different plasma location;

– at simultaneous operation of magnetron and plasma source the ion current to a sample is not an arithmetic sum of the currents from these devices;

– the sample shape demonstrates significant influence on the ion current in the researched system.

The obtained results are useful for investigation of effect of nonequilibrium heating of the sample surface due to relaxation of kinetic energy of ions, atoms and electrons, as well as energy of exothermic chemical reactions at synthesis of Ta_2O_5 and TaB_2 films.

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ИССЛЕДОВАНИЕ ВЗАИМОДЕЙСТВИЯ МЕЖДУ ИОННО-ПУЧКОВОЙ ПЛАЗМОЙ И ОБРАБАТЫВАЕМОЙ ПОВЕРХНОСТЬЮ ПРИ СИНТЕЗЕ ДИБОРИДА И ПЕНТАОКСИДА ТАНТАЛА

С. Яковин, А. Зыков, С. Дудин, А. Дахов, Н. Ефименко

Исследованы пространственные распределения плотности ионного тока на образец в технологической установке с магнетроном и индукционным источником плазмы. Измерены зависимости потока ионов на обрабатываемую поверхность от параметров процесса осаждения, таких как: мощность разряда магнетрона и индуктивного разряда, тип и давление газа, мощность, потенциал смещения, подаваемый на образец. Обсуждаются влияние неравновесного нагрева поверхности образца за счет релаксации кинетической энергии ионов, атомов и электронов, а также энергии экзотермических химических реакций при синтезе пленок Ta_2O_5 и TaB_2. Исследовано влияние формы образца на ионную бомбардировку.

ДОСЛІДЖЕННЯ ВЗАЄМОДІЇ МІЖ ІОННО-ПУЧКОВОЮ ПЛАЗМОЮ ТА ОБРОБЛЮВАНОЮ ПОВЕРХНЕЮ ПРИ СИНТЕЗІ ДИБОРИДУ ТА ПЕНТАОКСИДУ ТАНТАЛУ

С. Яковін, О. Зиков, С. Дудін, О. Дахов, Н. Єфименко

Досліджено просторові розподіли щільності іонного струму на зразок у технологічній установці з магнетроном та індукційним джерелом плазми. Виміряно залежності потоку іонів на оброблювану поверхню від параметрів процесу осадження, таких як: потужність магнетрона і індуктивного розряду, тип і тиск газу, потужність, потенціал зсуву на зразок. Обговорюється вплив нерівноважного нагріву поверхні зразка за рахунок релаксації кінетичної енергії іонів, атомів і електронів, а також енергії екзотермічних хімічних реакцій при синтезі плівок Ta_2O_5 і TaB_2. Досліджено вплив форми зразка на іонне бомбардування.