THE INVESTIGATION OF THE OPTICAL SPECTRA IN PROCESS OF MAGNETRON DEPOSITION

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We describe the state-of-the-art method of monitoring optical parameters the cylindrical gas discharge plasma of magnetron type. An analysis and characterization of the spectrum during a process of titanium nitride deposition is carried out. The optimum conditions of titanium nitride synthesis on substrates are determined. A characterization of deposited TiN films is made.

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1. INTRODUCTION

Magnetron sputtering systems represent well approved technology for a deposition of different functional films. However, a state of the art of PVD technologies for a magnetron deposition of thin films and a fast progress in development of nanotechnologies put forth higher demands to the functional characteristics of deposited films [1]. This, in turn, requires further development of both the systems themselves, and the technologies of application of respective plasma devices. In case of synthesis binary compounds by means of magnetron sputtering systems, the problem of monitoring the magnetron plasma is one of the top priorities [2].

In work [3] the first results of experimental studies of original cylindrical sputtering system of inverted type under conditions of sputtering binary compounds of titanium nitride and titanium dioxide, including their certain optical features, were presented.

This work represents further development of these researches with the use of modern optical device and the real-time method of monitoring optical parameters the cylindrical gas discharge plasma of magnetron type. Analysis and characterization of the spectrum in a process of titanium nitride deposition are performed. Optimum conditions of TiN synthesised are determined.

2. EXPERIMENTAL SETUP

Scheme of experimental setup is shown in Fig.1. A detailed description of the cylindrical type magnetron sputtering system is presented in [3]. We describe here only the main parameters of the system.

Cathode (3) of the magnetron was made of titanium and had a shape of hollow cylinder with 230 mm internal diameter and 140 mm height. It was mounted inside the vacuum chamber. System of permanent magnets (1) having shape of two combs was located behind the cathode. One comb was targeted to the cathode by their S poles and other by their N poles. Magnetic system created the arched field, which formed a closed meander-like path in the near-cathode surface with tangential strength 0.03–0.05 T. Anode system (4) consisting of 9 non-magnetic rods, 45 mm spacing from the cathode surface. For uniform sputtering of the cathode, special unit rotated the magnetic system and anode rods around the cathode axis with ~ 0.1 Hz. Magnetron discharge was ignited from the power source which stabilized the discharge current with value in a range of 5 – 18 A. Argon (Ar) with a pressure of $(2 – 5)\cdot 10^{-3}$ Torr was used as the working gas, and nitrogen (N_2) served as reactive one.

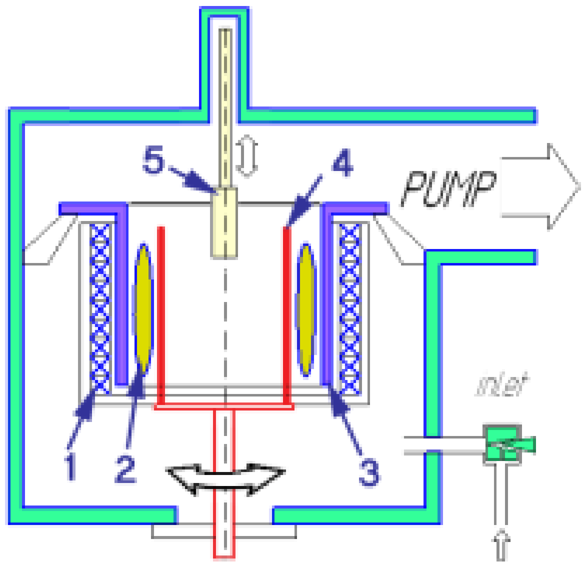

Fig.1. Scheme of experimental setup

The emission of the plasma discharge was led out of the vacuum chamber by means of tube with turning mirror. For protection of turning mirror from undesired coating, limiting aperture with 2 mm diameter was mounted at the end faced to the discharge. Spectrum of the plasma optical emission from magnetron discharge was analyzed by Plasma Spec device. Optical emission came to diffraction grating of the device, and obtained spectrum was read by CCD linear detector and was transferred via USB cable to the computer. Optical device Plasma Spec was assembled using common Czerny-Turner scheme and allowed spectrum readout time of 5 ms – 10 s with 0.6 nm resolutions in 350–820 nm wavelength range. Software supplied together with the device allowed monitoring of selected spectrum lines for a measured time, either with or without integration under line area, which is a very important for observation of dynamics of plasma sputtering processes. In these researches emission lines of argon and titanium atoms and nitrogen molecules were identified and separated. Analysis of the plasma emission spectrum dynamics depending on the sputtering process allowed determination the optimum conditions of stoichiometric films depositions. Obtained samples were subjected to micro-hardness analyses, and X-ray phase analysis.

3. EXPERIMENTAL RESULTS

In a process of analysis of obtained optical spectra of magnetron discharge plasma it was found that the spectrum contain 77 titanium lines, 41 argon lines, and 3 lines of molecular nitrogen under conditions of reactive sputtering. For observing the dynamics of spectrum variations, intensive and enough separately located lines of Ti (465,65 nm), Ar (696,54 nm) and N_2 (357,69 nm) [4] were selected. An area under the curve served as defining parameter during spectrum line observation. Software allowed monitoring of variation of these areas in real time depending on the system parameters.

It is found the behaviour of spectrum lines each group (Ti, Ar, and N_2) is similar. Curves of the integral intensities of Ar and Ti emission lines depending on the discharge current and Ar pressure are shown in Figs.2-3, respectively. One can see (Fig.2) that Ti line grow faster, than linear, depend on discharge current while Ar lines are almost linear. As can see from Fig.3 Ti line intensity changes slowly than Ar line respond to working gas pressure.

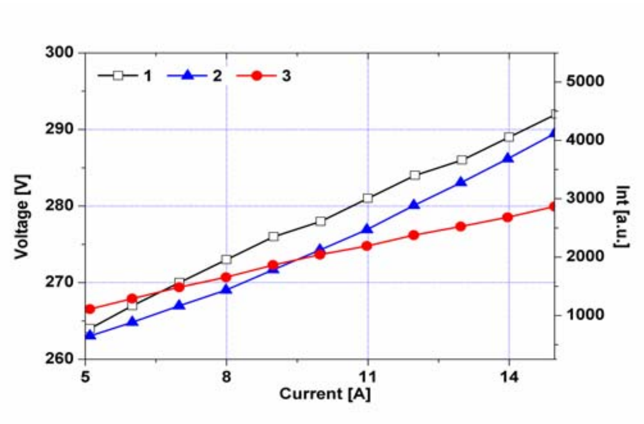

Fig.2. Dependence of discharge voltage and integral intensities of optical spectrum emission lines on discharge current [P(Ar)=5 mTorr]: 1 – discharge voltage, 2 – 465.65 nm (Ti), 3 – 696.54 nm (Ar)

Behaviour of integral spectra line intensity of magnetron plasma at reactive gas (in our case, N_2) supply into the chamber is shown in Fig.4. At this figure dependences of the discharge voltage on amount of reactive gas in the chamber are shown. The intensity of Ti line is more sensitive than N_2 line to reactive gas presence during practically important inlet interval of process. The TiN line intensity decreasing is due to the process of magnetron cathode spoiling during reactive sputtering [5]. Cathode spoiling takes place because of an active absorption of reactive gas on the cathode surface.

Absorption of reactive gas influences on N_2 line behaviour – its lines become visible only after almost complete spoiling of the getter surface that is, when excess of reactive gas takes place.

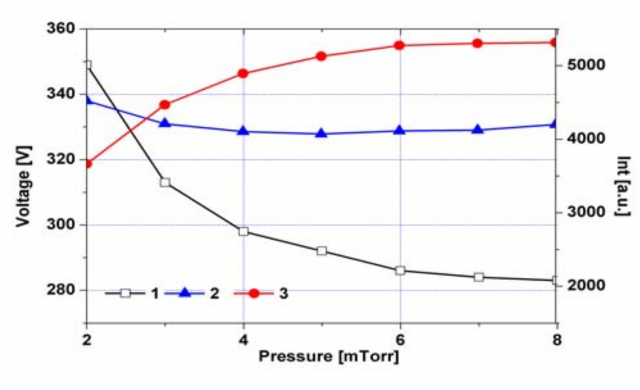

Fig.3. Dependence of discharge voltage and integral intensities of optical spectrum emission lines on Ar pressure [I=15 A]: 1 – discharge voltage,

2 – 465.65 nm (Ti), 3 – 696.54 nm (Ar)

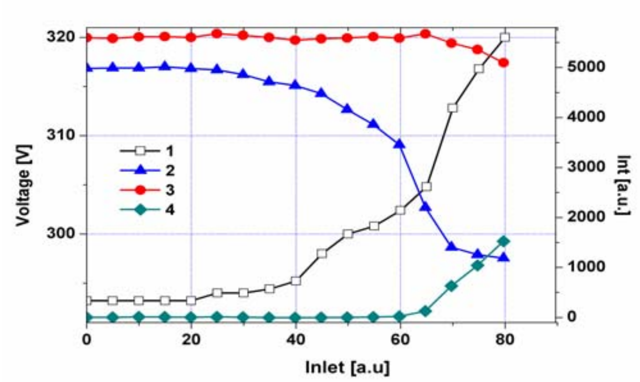

Fig.4. Dependence of discharge voltage and integral intensities of optical spectrum emission lines on nitrogen supply [P(Ar)=5 mTorr, I=15 A]: 1 – discharge voltage, 2 – 465.65 nm (Ti), 3 – 696.54 nm (Ar), 4 – 357.69 nm (N_2)

On behavior of an Ar line it is visible, that its intensity is reduced after there is a growth of intensity of a N_2 line. It is possible to assume, that the content of ions of Ar in an ion current on the cathode also reduced and result in slowing down of the cathode sputtering.

Working regime of the sputtering is defined by falling down range of the dependence for Ti lines, in vicinity of 65 a.u. of the reactive gas supply.

Such regime was used for deposition of TiN films. Results of X-ray structure studies are shown in Fig.5.

From comparison of relative intensities J (hkl)/ J (111) of X-ray diffraction lines of a powder of natural mineral TiN (osbornite) and TiN film on a glass substrate are visible, that the film has a preferred orientation of grains in a direction <111> normally to the surface of a substrate. The investigations have shown, that a film has micro-hardness from 17, 6 up to 22, 8 GPa. According to the data [6] on association of hardness on composition of TiNx, deposited film has composition 0, 92≤X≤1.

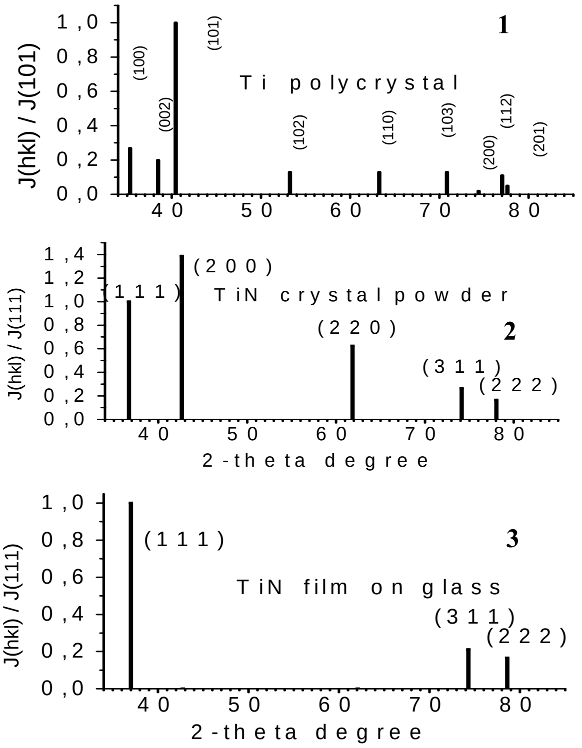

Fig.5. Identification of X-ray spectrum lines: 1 – polycrystalline titanium; 2 – polycrystalline titanium nitride; 3 – deposited sample of titanium nitride

5. CONCLUSIONS

This work presents the results of researches of optical spectrum of magnetron discharge plasma, and discusses the method of optical monitoring of deposition process of the films of binary compounds with titanium nitride taken as an example.

In spite of large quantity of observed plasma emission lines in optical spectrum range (about 100), it is sufficient to select one line from each group: for titanium, argon and reactive gas for the spectrum evolution monitoring. It is shown that the lines from different groups demonstrate different character of dependences under the change of experimental conditions.

In case of magnetron reactive sputtering argon lines are those to be used for controlling the discharge current value and working gas pressure. The sharp dependence on integral emission intensities of titanium lines with reactive gas supply makes this group of lines especially fit in the deposition process of the binary compound films. Low intensity of N_2 lines under working conditions makes inconvenient their use in technological applications.

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ИССЛЕДОВАНИЕ ОПТИЧЕСКИХ СПЕКТРОВ В ПРОЦЕССЕ МАГНЕТРОННОГО НАПЫЛЕНИЯ

А.В. Демчишин, А.Н. Евсюков, А.А. Гончаров, Е.Г. Костин

Предложен новый метод контроля оптических параметров плазмы цилиндрического газового разряда магнетронного типа в реальном масштабе времени. Проведен анализ и характеризация спектра в процессе напыления нитрида титана. Найдены оптимальные условия получения пленок нитрида титана. Выполнен их рентгенофазовый анализ и измерена микротвердость.

ДОСЛІДЖЕННЯ ОПТИЧНИХ СПЕКТРІВ В ПРОЦЕСІ МАГНЕТРОННОГО НАПИЛЕННЯ

А.В. Демчішин, А.М. Євсюков, О.А. Гончаров, Є.Г. Костін

Запропонований новий метод контролю оптичних параметрів плазми циліндричного газового розряду магнетронного типу в дійсному масштабі часу. Проведений аналіз та характеризація спектру в процесі напилення нітриду титана. Знайдені оптимальні умови отримання плівок нітриду титана. Виконано їх рентгенофазовий аналіз та виміряна мікротвердість.