STUDY OF NANOCRYSTALLINE TITANIUM DIOXIDE FILM SYNTHESIS IN A MAGNETRON-TYPE DISCHARGE BY MONITORING ITS OPTICAL AND PLASMODYNAMIC CHARACTERISTICS

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We present the results of experimental researches of plasmodynamic and optical characteristics of a magnetron-type cylindrical gas discharge. The study was carried out provided a permanent monitoring of the spectrum emitted by plasma in the range 350–820 nm. For the synthesis of binary compound TiO_2, we have determined conditions which can be ensured by a support of the intensity of spectral lines emitted by reacting components and plasma-forming gas. A possibility to control the conditions of the fabrication of a TiO_2 film with the use of both the spectral characteristics of a discharge plasma and a variation of the discharge voltage has been analyzed. Ellipsometric and spectral studies of nanocrystalline titanium dioxide films revealed the dependence of the refractive index of a film on the film thickness.

1. Introduction

Today, there is no doubt that thin films of titanium dioxide (TiO_2) with different modifications, owing to their unique electrophysical, optical, chemical, and bactericidal properties, are very promising for the advanced technologies of the 21-th century, first of all, for nanotechnologies. TiO_2 films are synthesized with the help of various chemical and physical vapor deposition techniques. Among them, the methods of magnetron sputtering have undoubted advantages from the viewpoint of the adaptation to the conditions needed for the synthesis of nano-sized films of binary compounds of chemically active metals [1].

In the previous work [2], a gas discharge generated in a dc inverted cylindrical magnetron was used to synthesize thin films of titanium nitride. The spectral monitoring of plasma parameters in real time was applied for the first time. This work is a development of the ideas proposed in work [2] for the fabrication of nanocrystalline TiO_2 films.

It is worth noting that inverted cylindrical magnetrons differ from direct ones in that, in the former, the internal surfaces of hollow cylindrical cathodes are sputtered. Coatings are deposited onto extended specimens of complicated shapes from various directions. This work aimed at studying the discharge parameters and the spectral plasma characteristics in an inverted cylindrical magnetron [3] and finding their relation with the partial pressure of oxygen, as well as optical properties of fabricated TiO_2 films.

2. Experimental Installation

A magnetron consists of a hollow cylindrical Ti cathode, a system of permanent magnets, and rotating rod anodes. At the cathode center, there is a specimen holder (cylindrical or prismatic).

The magnets create an arc magnetic field on the cathode surface with a tangential component of $0.03-0.05$ T. The arc field forms a closed meander-like curve above the cathode surface. The cathode erosion region is of the same shape. The magnet motion provides the uniformity of cathode sputtering and prevents from the formation of erosion cavities. This circumstance enhances the utilization ratio of a cathode substance up to 80%, which is an advantage in comparison with planar magnetrons. The second advantage is the stability of discharge parameters in time. In a planar magnetron, there emerge erosion cavities on the cathode with time. At those places, the plasma concentration increases, which gives rise to a change of current-voltage characteristics [4], a reduction of the discharge voltage and the ion energy, and variations of both the deposition rate and the coating thickness distribution. As a result, the reproducibility of the deposition process becomes violated, which requires an on-the-fly correction of the discharge current.

At the same time, the invariance of discharge parameters is especially needed at the reactive deposition of compound films, because the ranges of discharge voltage and reactive gas flow, which are required for the reproduction of film properties, are narrow. The position of this region directly depends on the discharge power and the geometric dimensions of the cathode and the surface to be deposited on [5].

The third advantage of an inverted magnetron consists in that its geometric configuration creates higher densities of the substance that is sputtered from the cathode on the substrate in comparison with that in planar magnetrons. The design of the chamber with a magnetron has been reported in work [6] in more details. Vacuum was obtained with the use of a high-vacuum turbo-molecular pump and a two-stage preevacuation unit with a Roots blower pump. The combination of those pumps allowed the evacuation rate be kept at a constant level up to 30 mTorr. The residual pressure in the chamber was $P \approx 4 \times 10^{-6}$ Torr. Argon was used as a plasma-forming gas, and oxygen as a reactive one. Separate channels were used for the gas supply through an inlet valve, with a precision (1%) manometer located before it.

A compact modern spectrometer Plasma Spec developed at the Institute of Physics of the NASU was used to monitor the deposition process. It allows the real time registration of a discharge plasma spectrum to be made. The time of the full spectrum record was 5 ms . The spectrum was registered by a charge-coupled device (CCD) line and processed, by using a special software. The total spectrum and selected spectral lines in the wavelength range $\lambda = 350 - 820$ nm were registered with an optical resolution of 0.6 nm. The spectrometer software allowed the selected spectral lines to be monitored depending on variable parameters of the sputtering process.

When the films of chemical compounds are deposited, it is necessary to find the deposition "working point" for a given compound (in our case, it is TiO_2) with respect to fluxes of plasma-forming and reactive gases and keep the system of gas puffing within the given parameter range. The check of the oxygen concentration with the use of vacuum gauges is complicated in this case, because the partial pressure of reactive gas is substantially (sometimes, by an order of magnitude) lower than that of plasma-forming gas, so it is impossible to resolve their contributions to the total pressure with a necessary accuracy. Therefore, an effective method for checking the concentration of reactive gas can be the monitoring of spectral intensities F of selected spectral lines. In addition, one can check the process of sputtering by analyzing the dependence of the discharge voltage U_P on the reactive gas flux (at a constant discharge current I_P).

The ellipsometry method [7] was used for the determination of optical parameters. Ellipsometric measurements were carried out on an LEF-3M-1 null-ellipsometer by varying the angle of light incidence from 44 to 80° with a step of 1–2° and at the wavelength $\lambda = 632.8$ nm. As a result of wide-angle ellipsometric measurements, we obtained the dependences of ellipsometric parameters Ψ and Δ on the light incidence angle and compared them with corresponding values calculated for the isotropic case "medium–film–substrate" at various values of the refractive index n and the film thickness. The other calculation parameters were given as known constant quantities. Experimental values were averaged over four measurement zones [8].

In the work, we report the results of our researches of the electrophysical parameters of discharge and the spectral characteristics of plasma in the inverted cylindrical magnetron. The researches were aimed at establishing the interrelation of those quantities with the oxygen flux and the optical properties of deposited TiO_2 films. We also considered a possibility of checking the conditions of TiO_2 film production by monitoring the discharge voltage.

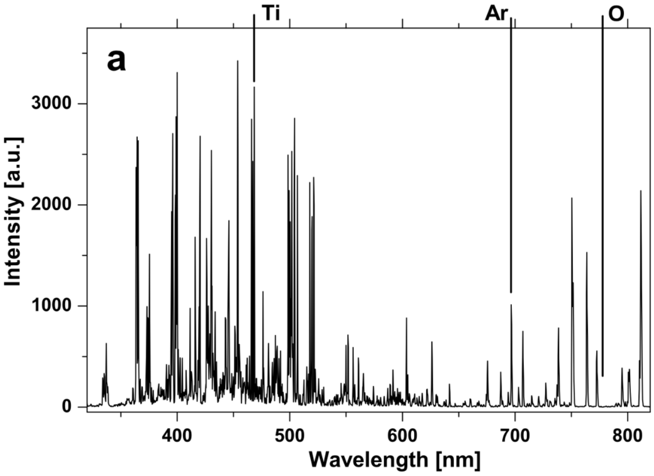
3. Results and Discussion

3.1. Discharge parameters

The spectral lines of radiation emission were identified on the basis of total discharge spectra. To resolve the overlapping lines (produced by Ti, Ar, and O_2), a glow discharge was ignited between auxiliary electrodes, when they were not practically sputtered, in the argon or oxygen environment in the chamber.

In Fig. 1, the spectra obtained at sputtering Ti (*a*) and TiO_2 (*b*) films are shown. When titanium dioxide is sputtered, the spectrum of magnetron plasma predominantly contains the lines of argon and oxygen. The lines of titanium have a very small intensity at the working point. The selected line of atomic oxygen ($\lambda = 777.19$ nm) is marked by an arrow. The dynamics of intensity variation of selected lines (four lines were watched simultaneously) was used to monitor the sputtering process and to check the stability of oxygen and argon puffing rates.

The Ar flux was monitored by its lines located not too close to Ti ones. For instance, the Ar lines with wavelengths of 430.01, 433.35, and 434.52 nm are close



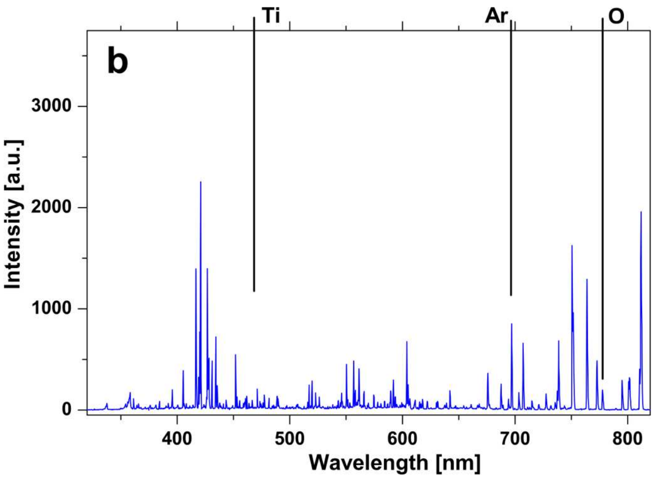


Fig. 1. Total spectrum of emission lines produced by discharge plasma in a magnetron at sputtering Ti (*a*) and TiO_2 (*b*) films

to the Ti lines with wavelengths of 430.05, 433.79, and 434.43 nm, respectively. In this case, the contribution given by the edges of a closely located Ti line, which decreases with the O_2 puffing, can result in a reduction of the Ar peak amplitude. The separately located Ti (468.2 nm) and oxygen (777.19 nm) lines were selected for the analysis.

In Fig. 2, the dependences of the intensity of some spectral Ti lines on the oxygen flux, provided that I_P is constant, are depicted. One can see that all Ti lines behave identically: their intensities diminish to a certain minimum with the growth of the O_2 flux (Fig. 2, curves *1* to *5*). The reduction of the intensities of Ti lines can be associated with a decrease of the metal atom concentration in the discharge gap. The decrease of the Ti atom concentration can be caused by at least two factors.

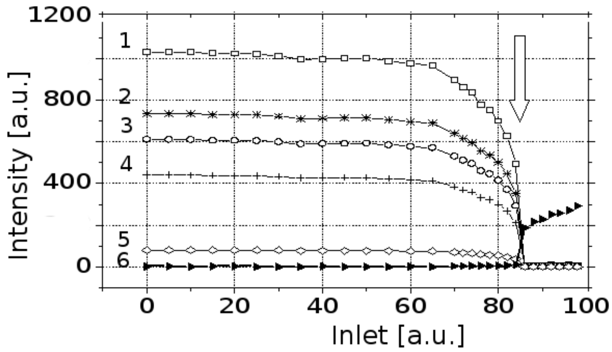


Fig. 2. Dependences of the radiation intensity on the oxygen flux for the Ti-lines at 468.2 (*1*), 501.4 (*2*), 521.04 (*3*), 375.3 (*4*), and 453.5 nm (*5*), and the O-line at 777.19 nm (*6*) at constant I_P

The first factor is the chemisorption of oxygen on the cathode surface and the formation of oxides on it. As a result, a fraction of the clean cathode surface, which Ti atoms are sputtered from, decreases. The number of Ti atoms reflected from the oxide-covered surface is small, because the coefficient of titanium sputtering from oxides is lower than that from metallic titanium. This corresponds to a higher Ti–O binding energy than the Ti–Ti one. According to the results of work [9], the sputtering coefficient amounts to 0.3 at./ion for Ti and 0.015 at./ion for titanium dioxide, provided that the energy of argon ions is 300 eV.

The second factor is the participation of oxygen ions in the cathode bombardment, i.e. a mass decrease of bombarding particles (in particular, the argon mass is 40 a.m.u., the oxygen one is 32 a.m.u.), and, as a consequence, a reduction of the Ti atom escape from the cathode surface.

At last, some decrease of the intensity of Ti lines (provided the same concentration of Ti atoms in plasma) can take place owing to electron energy losses which are spending for the excitation of electron-vibrational bands of molecular oxygen and its dissociation.

At low O_2 fluxes, the intensity of the atomic oxygen line (at 777.19 nm) is insignificant, but grows stepwise as the fluxes needed for the TiO_2 compound to be formed on the substrate (shown by an arrow in Fig. 2) are reached. No other oxygen lines, which would be more intense in the deposition zone, have been revealed in the spectrum.

The variation of the discharge voltage U_P during the oxygen puffing (in the regime of constant discharge current) demonstrates a peculiarity which can be used for monitoring the reactive deposition of TiO_2 films. The

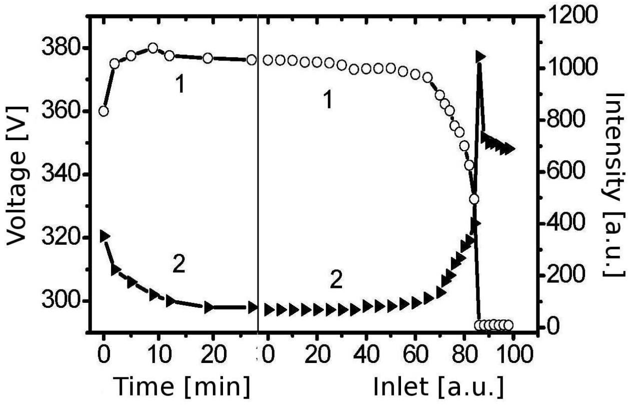


Fig. 3. Dependences of the Ti(468.2 nm) line intensity (*1*) and the discharge voltage U_P (*2*) on the time passed after the discharge ignition (on the left) and the oxygen flux Q (on the right). I_P = 15 A

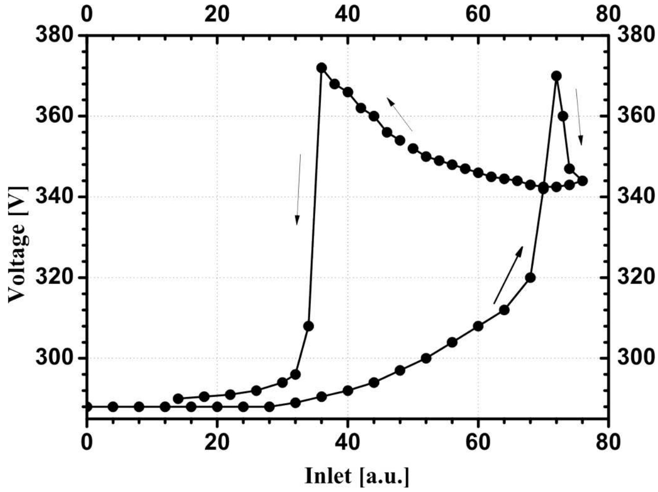


Fig. 4. Hysteresis in the dependence of the discharge voltage on the oxygen flux

reason for that is a considerable magnitude of this variation after the "working point" has been reached (see Figs. 4 and 5). At reactive sputtering, the discharge is governed by quantities that are given experimentally. These include the discharge current I_P, the argon flux Q(Ar), the reactive gas flux $Q(O_2)$, and the evacuation rate. The discharge voltage U_P, the partial pressures of gases, and the radiation emission intensities F of O_2, Ti, and Ar lines are functions of those quantities. Therefore, the quantity U_P, as a function of the reactive gas flux, can be used for monitoring the fabrication of films of a given compound at a constant discharge current.

In the framework of both checking methods (by F and U_P), it is necessary to achieve the limiting pressure of residual gases, in order that their background pressure be fixed and minimal, and to "train" the magnetron cathode before sputtering, by cleaning its surface from contaminants. This is related to the influence of background residual gases and surface contaminants of the cathode on U_P and F. In Fig. 3, the dependences of the quantities U_P and F(Ti) on the time, reckoned form the discharge ignition (at the cathode training), and the flux $Q(O_2)$ are shown.

One can see that, just after the plasma ignition, the discharge voltage is raised, whereas the radiation intensity F(Ti) is lowered, appreciably, in comparison with the accuracy of their stabilization at the "working point". In other words, impurities of residual gases affect both U_P and F(Ti) like a non-controllable additive of the reactive gas.

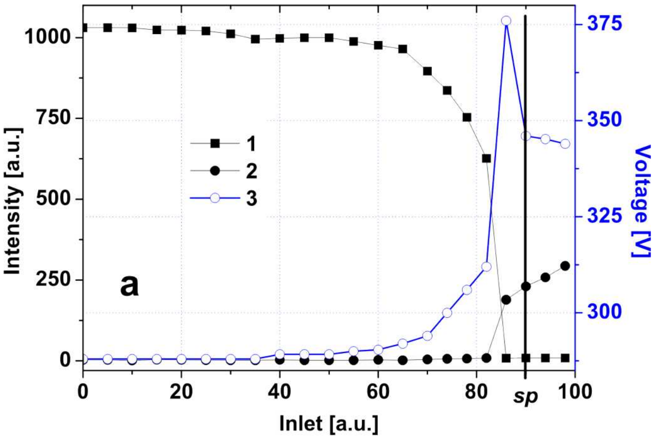
It was found that, in the cylindrical magnetron case, the dependences of the spectral line intensities and the discharge voltage on the O_2 flux have a hysteresis; i.e. those dependences are not the same at the increase and decrease of the O_2 flux. In Fig. 4, an example of hysteresis in the dependence of the discharge voltage on the oxygen flux is depicted. The arrows correspond to the direction of oxygen flux variation.

The hysteresis results from the difference between the coefficients of cathode sputtering in the "metal" and "oxide" states. At the beginning of the oxygen puffing process, when O_2 fluxes are low, the titanium cathode surface is slightly oxidized, because oxygen is effectively removed from the surface by ionic bombardment. It takes place until the cathode surface becomes covered with an oxide film, and the cathode passes into an "oxide phase".

In addition, the rate of Ti sputtering from oxides is substantially lower than that from the metal state, the coefficient of secondary ion-electron emission of oxidized Ti is less than that of metallic one (about 0.06 and 0.114, respectively) [8]. This circumstance gives rise to a reduction of the ion concentration in the discharge gap. A decrease of the charge carrier concentration induces the growth of the resistance across the discharge gap. A support of the discharge current by a constant one gives rise, in this case, to the growth of U_P at the O_2 puffing, as is shown in Fig. 4.

When the O_2 flux is reduced with the same rate, the sputtering of oxidized layers runs less effectively, so that the O_2 flux has to be decreased for the cathode to be switched back into the "metal" phase.

The dependences of U_P, F(Ti), and $F(O_2)$ on the reactive gas flux, which are presented in Fig. 5, can be compared with one another and with the composition



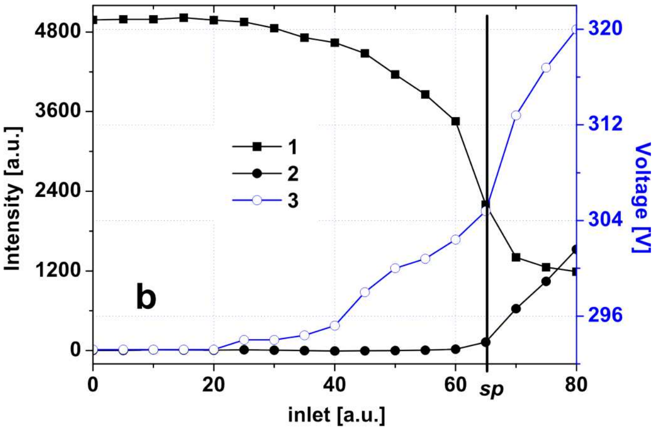


Fig. 5. (**a**) Dependences of the intensities F of the Ti(468.2 nm) (*1*) and O(777.19 nm) (*2*) lines and the discharge voltage U_P (*3*) on the oxygen flux. (**b**) Dependences of the intensities F of the Ti(468.2 nm) (*1*) and N(360.17 nm) (*2*) lines and the discharge voltage U_P (*3*) on the nitrogen flux. The discharge current is 15 A. The film deposition zones are shown by arrows

of films obtained. Note that, in contrast to analogous curves for the N_2 flux, presented in Fig. 5,*b*, the dependence of U_P on the O_2 flux (Fig. 5,*a*) is characterized by a more considerable voltage growth and the presence of a narrow maximum in the range of TiO_2 film saturation with the reactive gas.

In the region of the oxygen flux just before the deposition zone, which is indicated by arrows in Fig. 5, a drastic growth of U_P coincides with a faster decay of the intensity F(Ti). In the same interval, the intensity of the line O(777.19 nm) starts to grow, which is caused by an increase of both the partial pressure of oxygen and its dissociation into atoms. The growth of the partial pressure testifies to the saturation of a deposited film with oxygen (the formation of TiO_2) and a simultaneous reduction of the rate of oxygen absorption by this film.

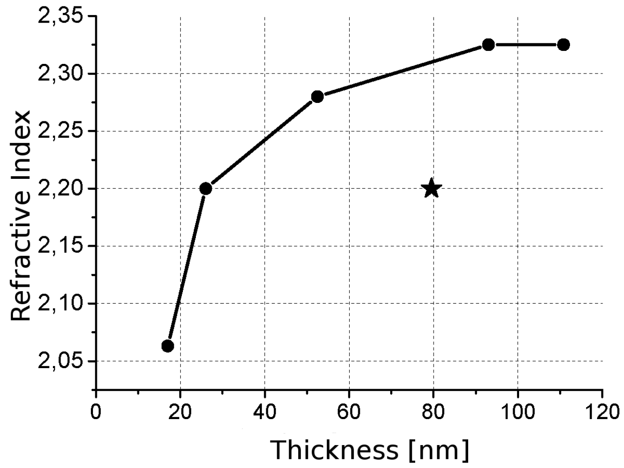


Fig. 6. Dependence of the refractive index of TiO_2 films on the film thickness at P(Ar) = 5 mTorr and I_P = 15 A. An isolated point corresponds to a film obtained at P(Ar) = 17 mTorr and I_P = 15 A

In the region, where the discharge voltage has a peak, the Ti line intensity falls down to an insignificant value, whereas the O_2 line intensity grows stepwise. The latter circumstance means that the saturation of the main bulk of a deposited film with oxygen occurs very quickly in this region of oxygen fluxes. After the film surface has been saturated with oxygen, the oxygen excess escapes into the discharge zone.

It should be noted that, should a less chemically active gas be used as a reactive one, the growth of the intensity of a reactive gas line would not be so drastic. For instance, in the case of TiN film sputtering, the intensity of the reactive gas line increases less sharply (Fig. 5,*b*). This is associated with the fact that titanium nitride is able to continue the nitrogen absorption after having reached the composition TiN_X, where $X \geq 1$. The compound TiN_X, with X ranging from 0.6 to 1.2, is thermodynamically stable, according to the data of work [7].

Since the intensity of Ti lines is weak in the working interval of the oxygen flux, the discharge voltage after the maximal point or the intensity of the atomic oxygen line after the section of stepwise growth was used to monitor the deposition of TiO_2 films. Note that the surface of those anodes, which are located closer than the specimen to the cathode that emits titanium atoms, remains conductive and capable of supporting the discharge burning.

3.2. Optical properties of films

In Fig. 6, the results of measurements of the TiO_2 film refractive index n for various film thicknesses are exhibited. The deposition rate of TiO_2 films was 5.5 nm/min. The specimens were obtained identically. Their thickness was determined by the corresponding deposition time. The films were deposited at such an O_2 flux, when the jump of the oxygen line intensity (see Fig. 5) had already occurred. Hence, the absorption of oxygen by the titanium film had already passed the saturation stage even at those surface sections, where the arrival rate of titanium atoms was maximal. (The total surface which was covered with the film also included the area of vacuum chamber walls.)

The maximal rate of titanium arrival onto the surface was reached for substrates located at the magnetron center. Therefore, the ratio between Ti and O_2 fluxes for specimens, which are described by curve *1*, corresponds to the formation of TiO_2 compound. (There is no information in the literature [10] concerning titanium oxides higher than TiO_2 even in the cases where the sputtering was carried out with a considerable excess of the O_2 flux.)

It is evident that a reduction of the film thickness, especially within the interval of 20–30 nm, gives rise to a substantial decrease of n. For the sake of comparison, note that, according to the data of work [11], the dimensions of nanocrystallites in TiO_2 continuous films sputtered onto an unheated substrate with the use of a similar magnetron sputtering technique amount to 17–24 nm, i.e. the minimal film thickness in Fig. 6 is comparable with the average crystallite size. Toward larger thicknesses, the curve demonstrates a tendency for the n-value to reach a plateau. But even at a thickness of 110 nm, n does not reach yet the value of refractive index for polycrystalline porous-free anatase ($n = 2.52$ at $\lambda = 550$ nm [12]).

Note that, if the film microstructure had remained invariable at a thickness reduction, the value of n should not be changed as well. A reduction of n testifies to considerable microstructural modifications in thin layers. Since the films were deposited at the same rate, it is impossible to suppose that they differ in the amount of contaminants absorbed from the residual atmosphere of a vacuum chamber. The absence of a substantial heating during the deposition excludes the diffusion of impurities from the substrate. The most probable explanation of the behavior of curve *1* is the model of temperature zones of film growth, which was proposed in work [13] and developed for the case of sputtering methods in work [14]. In those models of film growth, the major factor that influences the film microstructure is the "relative temperature" $T_s/T_{\rm fm}$, where $T_{\rm s}$ is the temperature of a substrate, on which the film grows, and $T_{\rm fm}$ is the melting temperature of a film material, both are measured in Kelvins. If the substrate temperature is low, $T_{\rm s} < 0.3T_{\rm fm}$, the surface diffusion of adatoms is also low, so that films with a low density, weakly determined grain boundaries, and grains separated by micropores grow. The melting temperature of TiO_2 is 2116 K, i.e. $0.3T_{\rm fm} = 635$ K. Such an equilibrium temperature of the substrate cannot be obtained, if the film is heated by discharge plasma at the deposition. In our experiments, it was about 475 K ($0.22T_{\rm fm}$). The time of temperature equilibration, when being measured with the help of a thermocouple, was about 9 to 10 min. At the same time, according to the results of work [15], where a film thermistor was applied, the temperature of the frontal substrate surface in the magnetron reaches the equilibrium value in 2.5 min.

All indicated above is enough for the explanation of why the refractive index in the obtained films is lower than that in polycrystalline solid anatase. For maximally thick films in Fig. 6, the growth temperature does not reach the value of $0.3T_{\rm fm}$ which is needed for a dense crystalline structure of TiO_2 to be formed. A decrease of n with a reduction of the film thickness (or the film growth time) corresponds to an even more lowered temperature of the film formation. For the thinnest films (of about 20 nm), the density and, accordingly, n can additionally decrease owing to a non-complete nanocrystallite coalescence at early film growth stages.

A specimen marked by a separate point in Fig. 6 was obtained at an argon pressure of 17 mTorr in the chamber which was 3.4 times as high as that for films with the same thickness on the curve. The corresponding refractive index n is lower. In the framework of model [14] which was developed for the film deposition by the cathode sputtering technique, the argon pressure is a parameter that shifts the "relative temperature" $T_{\rm s}/T_{\rm fm}$ toward larger values, i.e. the temperature range for the formation of films with lowered densities becomes shifted toward higher temperatures; and, if the temperatures are identical, the film density decreases with increase in the argon pressure. From the physical point of view, it can be explained by the scattering of Ti atoms by Ar ones, which is accompanied by energy losses. Therefore, Ti atoms become incapable of moving across the substrate surface due to the initial momentum, building into an ordered structure, and heating up the substrate at the thermalization of their kinetic energy.

The estimation of a free path of Ti atoms gives the values $L = 30.6$ mm for 5 mTorr and 9 mm for 17 mTorr. Therefore, since the distance between the cathode and the substrate is equal to 100 mm, the fraction of Ti atoms that reach the substrate without collisions amounts to 3.8% for 5 mTorr and a negligible value of about 1.5×10^{-3}% for 17 mTorr. A reduction of n with increase in the argon pressure was also observed in work [16]. When the argon pressure was varied (0.27, 0.8, and 2.7 Pa), the film refractive index changed as 2.42, 2.27, and 2.25, respectively.

Cylindrical inverted magnetrons are applied for the coating of three-dimensional specimens with complicated shapes which can include cavities. The results of our experiment testify that, when films with a sufficient thickness (> 40 nm) are sputtered, their refractive index n decreases by about 2% for cavity surfaces, where the sputtering at an angle of 45° took place.

4. Conclusions

By the spectra of plasma radiation in argon with the oxygen admixture in the wavelength range 350 − 820 nm, the lines of gas components and a substance sputtered from the cathode have been identified for a cylindrical gas discharge of the magnetron type. The influence of the reacting O_2 gas flux on the discharge voltage in an inverted cylindrical magnetron and the intensity of the radiation emitted by Ti atoms and molecules and atoms of the reacting gas has been studied.

By analyzing the variations of both the intensity of spectral lines and the discharge voltage, the conditions needed for the synthesis of binary TiO_2 compounds have been determined. The optical monitoring of spectral lines turned out to be more informative, because it allows the behaviors of several components of a gas medium in the discharge volume to be watched simultaneously.

The dependence of the refractive index of TiO_2 films on their thickness – namely, its reduction in films with nano-sized thicknesses, which is connected with structural features of the film growth – has been revealed. Those features can be determined more exactly with the help of electron microscopy methods which will constitute the purpose of our further researches.

Using TiO_2 films as an example, the capability of the reactive deposition of films of insulating materials in a dc inverted cylindrical magnetron has been demonstrated. Neither high-frequency power sources nor complicated devices for their matching with a discharge system are required at that.

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ВИВЧЕННЯ ПРОЦЕСУ СИНТЕЗУ НАНОКРИСТАЛІЧНИХ ПЛІВОК ДВООКСИДУ ТИТАНУ В РОЗРЯДІ МАГНЕТРОННОГО ТИПУ ЗА ЙОГО ОПТИЧНИМИ ТА ПЛАЗМОДИНАМІЧНИМИ ХАРАКТЕРИСТИКАМИ

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Р е з ю м е

Наведено результати експериментального дослідження плазмодинамічних і оптичних характеристик циліндричного газового розряду магнетронного типу в умовах безперервного контролю спектра, випромінюваного плазмою в діапазоні 350–820 нм. Визначено умови для синтезу бінарної сполуки TiO_2, які забезпечуються підтримкою величини інтенсивності спектральних ліній реагуючих компонентів і плазмоутворюючого газу. Розглянуто можливість контролю умов одержання плівок TiO_2 як по спектральних характеристиках плазми розряду, так і по зміні розрядної напруги. Еліпсометричні дослідження нанокристалічних плівок двооксиду титану показали наявність залежності показника переломлення від товщини плівки.