ТЕХНИЧЕСКИЕ НАУКИ
И ТЕХНОЛОГИИ

МАТЕРИАЛТАНУ
МАТЕРИАЛОВЕДЕНИЕ
MATERIALS SCIENCE

DOI 10.51885/1561-4212_2022_4_30
МРНТИ 61.74.31; 67.15.43; 67.15.55; 81.91.51

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STUDY OF MAGNETRON DEPOSITION AND IMMERSION COATING METHODS OBTAINING VANADIUM PENTOXIDE THIN FILMS

МАГНЕТРОНДЫ ТҰНДЫРУ ЖӘНЕ ИММЕРСИЯЛЫҚ ЖАБЫНДАРДЫ ЖАҒУ ӘДІСТЕРІМЕН ВАНАДИЙ ПЕНТОКСИДІНІҢ ЖҰҚА ҚАБЫРШАҚТАРЫН АЛУДЫ ЗЕРТТЕУ

ИССЛЕДОВАНИЕ МЕТОДОВ МАГНЕТРОННОГО ОСАЖДЕНИЯ И ИММЕРСИОННОГО НАНЕСЕНИЯ ПОКРЫТИЙ С ПОЛУЧЕНИЕМ ТОНКИХ ПЛЕНОК ПЕНТОКСИДА ВАНАДИЯ

Abstract. *In this work, thin coatings of vanadium oxide were obtained by magnetron deposition, immersion coating with different chemical composition of the initial solutions. Thermochromic and electrochromic behavior of thin-film coatings made of vanadium oxide causes great interest in this material in the smart windows industry. The methods of widely used deposition methods for obtaining vanadium pentoxide films were investigated, the basic physico-chemical properties of the coatings obtained were studied. As a result of research in the composite structure, semiconductors with VxOy demonstrate additional characteristics, which increase the scope of their application, including in nanophotonics and photovoltaics. The optical properties of nanoscale particles and quantum dots of vanadium oxide have well-adjustable parameters, which, along with the use of the surface plasmon resonance effect, endow the material with many advantages. The phase composition and crystal structure of the obtained samples were investigated by X-ray diffractometer X'pert PRO (PANalitical). The optical transmission and absorption spectra of the formed thin films were measured on a double-beam UV/Vis Specord plus spectrophotometer (Analytik Jena), while the cell position for the sample under study is located directly in front of the detector, which allows the analysis of cloudy samples.*

Keywords: *vanadium oxide; smart glasses; thermochromism; phase-transition materials; thermal properties; composition.*

Аңдатпа. *Бұл жұмыста ванадий оксидінің жұқа жабындары магнетронды тұндыру, бастапқы ерітінділердің әртүрлі химиялық құрамымен батыру арқылы алынды. Ванадий оксидінің жұқа пленкалы жабындарының термохромды және электрохромды әрекеті smart windows индустриясында бұл материалға үлкен қызығушылық тудырады. Ванадий пентаоксидінің пленкаларын алу үшін кең қолданылатын қолдану әдістері зерттелді, алынған жабындардың негізгі физикалық-химиялық қасиеттері зерттелді. Композиттік құрылымдағы зерттеулер нәтижесінде VxOy бар жартылай өткізгіштер қосымша сипаттамаларды көрсетеді, бұл олардың*

қолдану аясын, соның ішінде нанофотоника мен фотовольтаиканы арттырады. Наноөлшемді бөлшектер мен ванадий оксидінің кванттық нүктелерінің оптикалық қасиеттері жақсы реттелген параметрлерге ие, олар беттік плазмалық резонанс әсерін қолданумен қатар материалға көптеген артықшылықтар береді. Алынған үлгілердің фазалық құрамы мен кристалды құрылымын X'pert PRO (PANalitical) рентгендік дифрактометр зерттеді. Құрылған жұқа пленкалардың оптикалық өткізу және сіңіру спектрлері specord® plus (Analytik Jena) екі сәулелі UV/Vis спектрофотометрімен өлшенеді, зерттелетін үлгі үшін ұяшықтың орналасуы детектордың алдында орналасқан, бұл Бұлтты үлгілерді талдауға мүмкіндік береді.

***Түйін сөздер**: ванадий оксиді; smart-шыны; термохромизм; фазалық өтпелі материалдар; жылу техникалық қасиеттері; композиция.*

***Аннотация**. В данной работе тонкие покрытия оксида ванадия были получены методом магнетронного осаждения, нанесение покрытий погружением с различным химическим составом исходных растворов. Термохромное и электрохромное поведение тонкопленочных покрытий из оксида ванадия вызывает огромный интерес к этому материалу в индустрии smart windows. Были исследованы способы широкоиспользуемых методов нанесения для получения пленков пентаоксида ванадия, изучены основные физико-химические свойства полученных покрытий. В результате исследований в композитной структуре полупроводники с VxOy демонстрируют дополнительные характеристики, что увеличивает области их применения в том числе в нанофотонике и фотовольтаике. Оптические свойства наноразмерных частиц и квантовых точек оксида ванадия имеют хорошо подстраиваемые параметры, что наряду с использованием эффекта поверхностного плазмонного резонанса наделяют материал многими преимуществами. Фазовый состав и кристаллическая структура полученных образцов исследованы рентгеновским дифрактометром X'pert PRO (PANalitical). Спектры оптического пропускания и поглощения сформировавшихся тонких пленок измерены на двухлучевом UV/Vis спектрофотометре Specord® plus (Analytik Jena), при этом положение ячейки для исследуемого образца находится непосредственно перед детектором, что позволяет анализировать мутные образцы.*

***Ключевые слова**: оксид ванадия; smart-стекла; термохромизм; фазопереходные материалы; теплотехнические свойства; композиция.*

Introduction. Thermochromic materials are the most important and fundamental components needed to achieve the thermochromic function for smart windows. Among the many thermochromic materials known, metal oxides, such as vanadium oxide, tungsten oxide, titanium oxide, and other elements are a major category.

It is known that in vanadium (IV) oxide at 67^0C the "semiconductor-metal" phase transition occurs, characterized by a sharp change in resistivity and light energy transmission coefficient in infrared and ultrahigh frequency ranges [1,2]. In this case, there is a jump of conductivity from 10-6 to 10-1 ohm*meter. The optical properties also change the refractive index decreases from 2.5 to 2.0 [3]. The change in the crystal structure is associated with the phase transformation of vanadium oxide from monoclinic to tetragonal, possessing the effects of resistive electrical switching, phase transition, photo-, electro-, and thermochromism. Thus, the unique phase-transition properties of vanadium (IV) oxide are used in the production of vanadium bronzes, as a semiconductor material for the thermistor, memory switches, displays, for glass coatings that block infrared radiation. Polycrystalline VO_2 films are used in electronic devices, infrared (IR) imaging devices [4], and non-linear optical radiation limiters, as media for hologram recording, in mirrors with controlled reflection coefficient.

This work is aimed at creating the basic foundations of thermochromic energy-efficient compositions based on vanadium oxide for use in the glass industry with the ability to transmit light, control heat and light energies in civil construction.

In Kazakhstan, there is no domestic production of thermochromic energy-efficient compositions based on vanadium oxide [5], and scientific research on their composition is not being carried out.

The purpose of this work is to develop a composition based on vanadium oxide, which has thermochromic energy efficient properties and is used in the glass industry for smart windows. The development of the composition will create favorable conditions in green building, while economically important is the reduction of high energy consumption due to urban development, population growth and improvement of indoor climate comfort requirements in civil engineering.

The main research approaches consist of the world-known methods of obtaining thermochromic fillers [6-9]; the available productive results of foreign research are based on methods of obtaining thermochromic fillers based on low molecular weight organic compounds, emissive polymeric or nanocomposite films, photo- and thermofunctional complexes of transition metals. Based on technological and economic analysis, the most optimal and technologically acceptable method for obtaining thermochromic compositions is the doping of films with phase-transition materials that allow controlling the main parameters of energy saving and other thermal properties, followed by application on glass and/or other substrates.

Therefore, there is a need to obtain thermochromic materials with low cost, suitable for use in the enclosing structures of buildings, to regulate the heat in their premises. According to the literature review, at present, the research aimed at the development of thermochromic materials based on vanadium oxide seems relevant [10-13]. VO_2 is considered to be the most studied and having a convenient phase transition temperature for measurements. For example, for VO_2 at 67^0C (340 K), the phase transition from semiconductor to metallic state occurs, depending on the impurity components and non-stoichiometry on oxygen. This oxide in the composition has a reflective effect. The phase transition in VO_2 can be considered superfast, since under essentially nonequilibrium conditions, for example, when exposed to a short pulse of light of sufficient energy, the transition time from the semiconductor phase to the metallic phase is about 100 ms [14-16]. This process is accompanied by a change in the optical characteristics of VO_2: reflection, absorption and transmittance coefficients. The transmission coefficient depends on the length and time interval of radiation and its polarization in crystallographic axes of VO_2.

Thus, vanadium oxide can be used in temperature sensors [17], optical switches [18-20], and glass coatings, as a carrier of information (due to the short transition time, 30 nanoseconds, and low excitation energy) [21].

Research methods. The works of domestic and foreign scientists devoted to the development of composite materials based on oxide materials will also be the methodological and theoretical base of the research[21-24].

The optimal conditions for the process of the formulation will be established in the laboratory based on the study of the physical and chemical characteristics of raw materials and products, namely strength, steadiness, chemical and radiological analysis, the dependence of changes in electrical resistance and optical transmission from temperature, thermal conductivity, degree of transmission of IR and UV radiation, moisture and chemical resistance, heat transfer coefficient, microhardness, the economic low cost is calculated, etc.

The crystallite growth of polycrystalline films made by simultaneous variation of sprayed vanadium oxide film thickness and time of heat treatment at different temperatures will be investigated using electron microscopy techniques. The study of the film structure will be carried out with an X-ray microprobe analyzer.

The main method to analyze the results of the research will be the methods of optical spectroscopy, the following equipments will be used: spectrophotometers SPECORD with built-in software and computer interface to determine absorption spectra, computer program MatLab for contour analysis and search for spectral forms of vanadium oxide and alloying additives, determination of the structure of absorption bands of isoptic densities.

Results and discussion. V_2O_5 thin film deposition technique.

This section presents the methods of obtaining V2O5 thin films. The results of surface morphology, structural properties, thermal characteristics, elemental composition and optical characteristics of the obtained materials are given.

1. Magnetron deposition of V_2O_5 thin films on ITO conductive glass.

For magnetron sputtering of thin films, the magnetron sputtering unit MAGNA TM-200-1 was used, the scheme of its working process is shown in Figure 1.

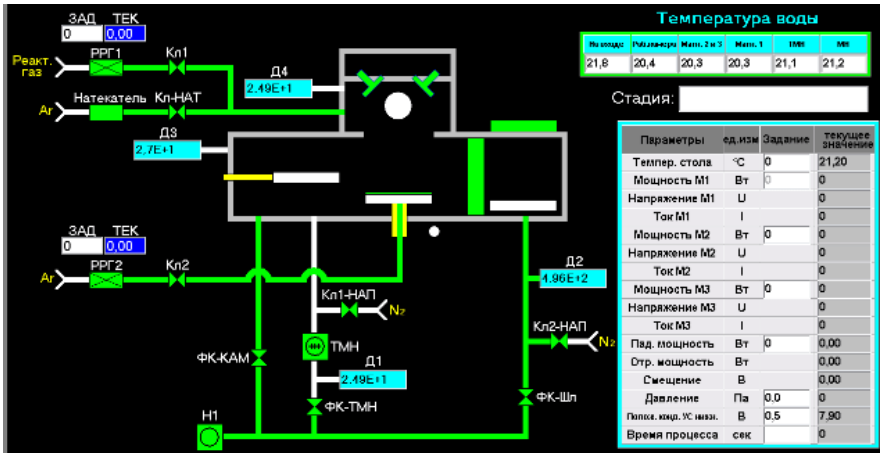


Figure 1. Installation diagram for applying metal layers

The formation of vanadium oxide thin films on the surface of ITO glass by magnetron deposition proceeded in the mode of energy source operation at a constant current. The deposition process was accompanied by feeding a mixture of working gases argon (99.95%) and oxygen (99.92 %) into a chamber with a deep vacuum ($2.95*10^{-5}$ millibar). The removal of oxides from the surface of the metal target was achieved by pre-spraying the vanadium target material in argon plasma for 15-25 minutes. Physical atomization of vanadium was carried out by bombarding the surface of the vanadium target with accelerated ions of atomizing argon gas, the purity of which was 99.98 %, followed by the reaction of vanadium atoms torn from the surface of the target with reactive gaseous oxygen. The supply of argon and oxygen gases was carried out through channels of independent gas intakes with controllers of a mass gas flow meter.

The process of magnetron deposition of vanadium oxide on the driving glass was started when the argon gas pressure reached 0.6 Pascale, with the reaction gaseous oxygen being supplied until 10 % of the argon in the reactor chamber was reached.

The power of the magnetron varied in the range of 100-500 watts. Sample 1 was formed when the magnetron power reached 100 watts, 2 was formed when the magnetron power reached 300 watts and 3 was formed when the magnetron power reached 500 watts. The substrate temperature was maintained at 300°C. The pumping rate was 0.6 l/hour, while the argon consumption was about 5.5 cm^3/hour. The obtained samples were analyzed by Raman spectroscopy (ACM - Raman - SBOM - TERS). Figure 2 shows the Raman scattering spectra of films 1-3 V_2O_5.

The traceable peak at 145 cm^{-1} is associated with vibrations of the V –O –V group. However, the poorly traceable peak does not characterize the V_2O_5 structure as a whole, showing only the relation of the film to the layered structures. The arrows in Figure 2, (a) show the more intense peaks whose Raman shift is characteristic of V_2O_5 orthorhombic phase structures – 283 cm^{-1}, 305 cm^{-1},525 cm^{-1}, 695 cm^{-1}, 987 cm^{-1}. When the magnetron sputtering power was increased to 300

W and 500 W, the intensity of the peaks belonging to the orthorhombic phase of V_2O_5 decreased, which probably refers to the change in the valence state of V_xO_y.

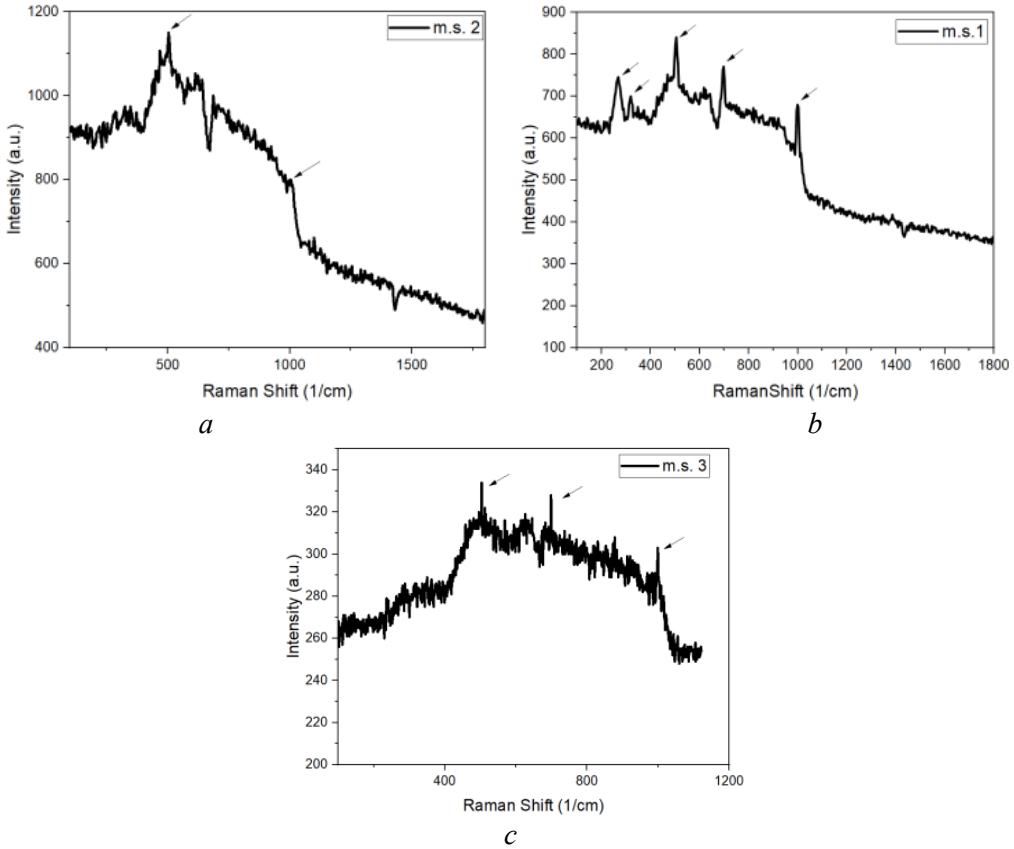


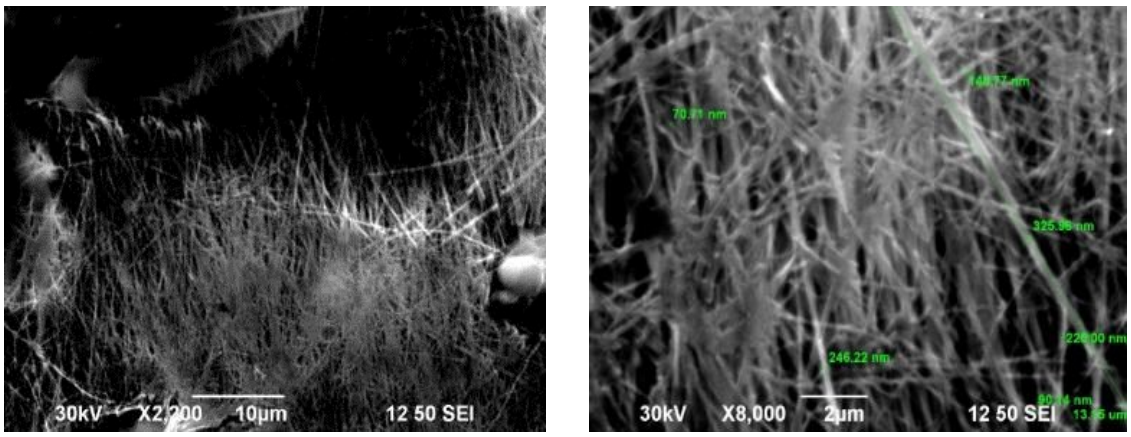
Figure 2. Raman scattering spectra of $V_2O_5$1-3 (a-c) samples obtained by magnetron sputtering

2. Hydrothermal synthesis of VxOy nanostructures. NH_4VO_3 aqueous solution was used for hydrothermal synthesis of VxOy nanostructures. After achieving complete dissolution of the substance in deionized water, the mixture was placed in a high-pressure autoclave reactor for hydrothermal synthesis. The temperature parameters of the synthesis were controlled by a muffle furnace. After several experimental works, the temperature of the working process was chosen in a range of 190 - 230ºC. The synthesis time varied from 24 to 36 hours. After the completion of hydrothermal synthesis, the samples were thoroughly cleaned in water to exclude residual products on the surface of the nanostructures. Filtration of the obtained material was performed to separate the synthesized material from the working solution. Heat treatment was carried out in two stages. The first stage is drying in an air atmosphere at a temperature of 100 °C to remove water for 30 minutes. The second stage was calcification at 400ºC to produce vanadium oxide fibers. The duration of the second stage was 120 minutes.

Details: 75 mL of an aqueous solution containing 50 g NH_4VO_3 was placed in a 100 mL autoclave reactor. A complete filling of the reactor with the solution was excluded, otherwise, a complete depressurization of the reactor would occur due to high pressures.

The surface morphology of the obtained samples was analyzed on the JSM-6490 LA (JEOL) and MIRA 3LMU (Tescan, Czech Republic) electron scanning microscope facilities with a direct-fired tungsten cathode. The main electron beam is generated by a heated tungsten filament or field

ejection gun and is usually accelerated by applying a voltage of 1-30 kV. The presence of electromagnetic lenses causes the beam to focus on the sample to a spot size in the nanometer range. When the electron beam penetrates the sample, several processes occur, such as absorption, scattering, and emission. The surface morphology of the obtained samples was studied by analyzing data from a secondary electron detector. Secondary electrons are generated when incident beam electrons with sufficiently high energy hit the sample. Since the energy of the secondary electrons is quite low (<50 eV), it is only possible to obtain information concerning the surface of the material. Back-scattered and diffracted electrons can also be detected (such electrons lose only 20-40% of their initial energy), but they appear at different diffraction angles. Micrographs of the nanofibers VO are shown in Figure 3.



a *b*

Figure 3. SEM image of the surface morphology of thin films

The figure shows images of the sample synthesized by the hydrothermal method for 20 hours. When analyzing the micrographic images (Fig. 3 a-b), we can see that when magnifying a powder particle with a diameter of several millimeters (Fig. 3, a), the nanofibers VOs, the length of which reaches several micrometers, while the transverse diameter varies from tens to hundreds of nanometers, i.e. the dimensions of nanofibers VOs lie in the submicron to nanoscale region. According to the results of the study of the surface morphology of the obtained materials, it was found that the permissible temperature of hydrothermal synthesis of nanofibers VO is the level of 220-240^{o}C. At the same time, the time parameters do not contribute characteristically to the development of the nanofibers VO morphology (Fig. 3, b). Thus, it was found that with insufficient time of hydrothermal synthesis, the structures of the material are not formed in the form of nanofibers VO, when the 24-hour synthesis is reached, nanofibers VO is traced in the volume of the solution (reactor).

When the synthesis time is increased up to 36 hours, the formed nanofibers VOs are deposited on the reactor walls, forming a powder-like mass. When studying the surface morphology of such a powder-like mass, it was noticed that the nanofibers VOs do not change the previously acquired geometric parameters, which indicates that there is no need for a long (more than 24 hours) hydrothermal synthesis.

Thermal characteristics of the samples obtained by the hydrothermal method were studied on a differential thermal analyzer unit with thermogravimetric analyzer attachment, DSC, with a measurement range $^{+}$/- 1000 mg with a resolution of 0.2 mg or 0.02 mg. The instrument is a metal resistor furnace with a temperature range from room temperature to 1600^{0}C with a scanning speed

of 0.01 to 100 °C/min. The main stages of mass loss of the test sample are demonstrated at 75°C, 650°C, 780°C and 960°C. The first stage of mass loss is explained by the phase transition, which gives the material special characteristics associated with the thermochromic properties of nanofibers VO, further mass loss is explained by possible calcification of the sample.

3. Spin-coating method of forming planar films on a conductive substrate. Spin-coating of planar V_2O_5 thin films uses a sol-gel technique. The installation for spin-coating consists of a speed-controlled rotating platform on which the sample is placed. The rotation speed of the platform with the sample varies from 500-4000 rpm. The positioning of the sample (ITO glass) on the surface of the platform should ensure that the sol prepared in advance hits the surface of the substrate. If a conductive glass is used as a substrate, the glass should be positioned so that the ITO layer is facing upwards to apply the working solution to its surface.

The recipe for the preparation of the solution for the spin-coating of V_2O_5 nanolayers consists of the preparation of a sol-gel. The sol consists of colloidal V_2O_5 obtained by rapid oxidation in an H_2O_2 medium. Recipe for the solution: 6 g of V_2O_5 powder was diluted in 60 ml of 36% technical H_2O_2 under rapid stirring in a cool environment (domestic refrigerator, 7°C) for three days. As a consequence of the above steps, the solution turns a dark red color. The viscosity of the solution depends on the stirring speed. The use of viscous solutions, when applying layers of V_2O_5 by spin-coating, does not allow obtaining uniform films.

When applying 10 layers of V_2O_5 in the spin-coating mode at 2500 rpm, the geometric cross-sectional size of the resulting film reaches 2.65 micrometers.

The optical transmission properties of electromagnetic waves in the spectrum from 300 to 1100 nm inherent to the samples formed at 3 and 5 cycles of V_2O_5 deposition in the spin-coating mode are shown in Figure 4.

Optical transmission spectra of V_2O_5 3 spincoated and V_2O_5 5 spincoated samples; are shown by black and red curves, respectively. It can be seen that 5 spincoated shows transmittance of the whole spectrum at the level of less than 1.5%, on the other hand, 3 spincoated sample has an optical transmittance level of more than 15%. Thus, it can be assumed that effective thermochromic and electrochromic properties are demonstrated by films whose transverse size is less than 2 micrometers.

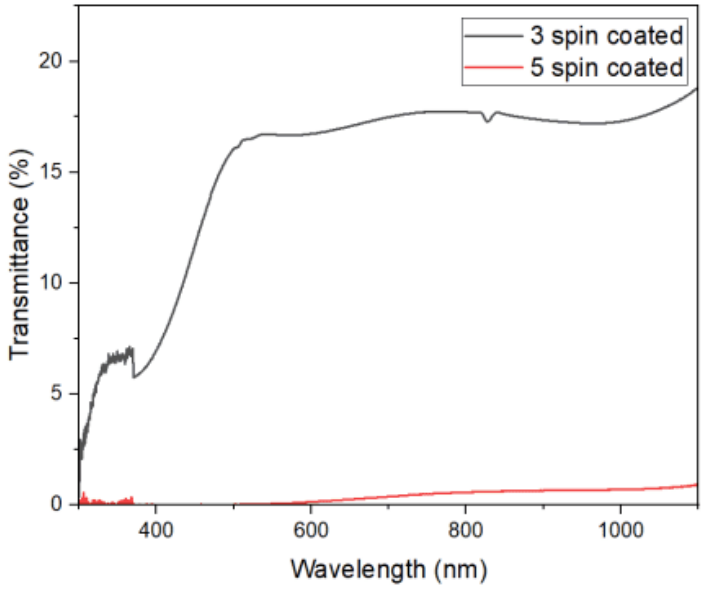


Figure 4. Optical transmission spectrum of samples 3 spin coated and 5 spin coated

4. Dip-coating method for obtaining planar coatings V_2O_5. Dip-coating method for the formation of V_2O_5 thin films was carried out on the automated installation shown in Figure 5.

Figure 5. Photographic image of the installation for carrying out dip-coating synthesis

The operating mode of the installation of the production by Nadetech Innovation is experimentally selected according to the following parameters:

- speed of substrate immersion in the solution is 50 mm/minute.
- substrate removal speed from the solution is 10 mm/min.
- substrate radial movement speed between vessels with different solutions 10°/sec.

The formation of V_2O_5 thin films by dip-coating thin layers was carried out by observing the following synthesis parameters: the conductive glass plate was consistently immersed in two containers. The first container contained the working sol-gel solution prepared according to the procedure of Section 1.3. The second one contained deionized water. When immersing the substrate into the first container, the reaction of V_2O_5 adsorption took place. The immersion time was 10 seconds. The second step of dip-coating synthesis was rinsing in deionized water for 20 seconds, resulting in the removal of excess material loosely bound by the substrate surface. Thermal treatment at 400 ℃ allows calcification and annealing of the substance.

The number of dip-coating cycles affects the optical properties of the resulting coatings. Thus, Figure 6 shows the absorption spectrum of V_2O_5 films at 1 and 5 cycles of dip-coating. The spectrum is recorded in the wavelength range between 300 and 1100 nm.

It can be seen that the absorption of light caused by the semiconductor material in the violet region dominates at 5-fold absorption of the solution. Whereas at 1x application of the product, the light absorption is reduced by two times.

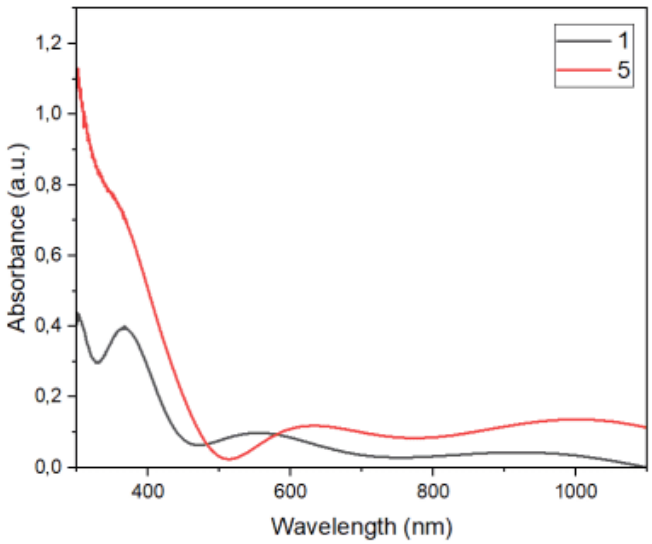


Figure 6. The absorption spectrum of V_2O_5 films at 1 and 5 dip-coating cycles

*Conclusion***.** Low-dimensional V_2O_5 films were grown on the surface of ITO glass by magnetron sputtering, sol-gel spin/dipcoating techniques, and hydrothermal synthesis. After the synthesis work, the material was post-processed, including annealing and calcification at different temperature ranges. UV methods/Type of spectroscopy studies of the optical properties of the samples were carried out. Structural studies were carried out using Raman spectroscopy. Thermal properties were studied by thermogravimetric analysis.

Acknowledgements. This research has been funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan (Grant No. AP09058456).

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