IRSTI 29.03.31 https://doi.org/10.26577/RCPh.2024v90i3-05

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EVOLUTION OF STRUCTURAL, OPTICAL PROPERTIES AND ELECTRICAL TRANSPORT Of NiO_x FILMS ANNEALED IN DIFFERENT ENVIRONMENT

The influence of the annealing medium of thin films of nickel oxide (NiO_x) on structural, optical and electrical transport properties is studied in this paper. NiO_x films were synthesized by sol-gel method and annealed at a temperature of 300 °C in air, inert nitrogen (N_2), oxygen (O_2) gases and vacuum. The values of the films thickness were determined from the transverse cleavage of the SEM image. The films thickness depends on the annealing medium. Measurements of the EDX spectra of NiO_x films were carried out. Analysis of the quantitative O/Ni ratio showed that the film annealed in an air atmosphere is close to the stoichiometric parameters of NiO. Measurements of the EDX spectra of NiO_x films were carried out. According to the Raman spectra, an increase in peak intensities is observed indicating an increase in the density of defects in the film by Ni vacancies depending on the annealing medium. Absorption spectra and Tauc plots of NiO_x films were obtained. The optical density of the films depends on the annealing conditions of the films. Data analysis shows that the band gap width depends on the annealing medium and varies from Eg = 3.12 eV to Eg = 3.45 eV. The analysis of the impedance spectra allowed us to estimate the values of the resistances R_1 and R_2. It is shown that an increase in the resistance of R_1 is associated with a decrease in the density of defects in the NiO_x film, decrease in the recombination resistance of R_2, and an increase in recombination processes at the interface.

Key words: nickel oxide, sol-gel technology, annealing environment, optical band gap, absorption coefficient, dispersion index, electrical transport characteristics.

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Әртүрлі ортада күйдірілген NiO_x қабықшаларының құрылымдық, оптикалық және электрлік тасымалдау қасиеттерінің эволюциясы

Бұл жұмыста никель оксидінің (NiO_x) жұқа қабықшаларын құрылымдық, оптикалық және электр тасымалдау қасиеттері зерттелді. NiO_x қабықшалары золь - гель әдісімен синтезделіп, 300 °C температурада ауада, инертті азот (N_2), оттегі (O_2) газдарында және вакуумда күйдірілді. Қабықшалар қалыңдығының мәндері СЭМ суреттеріндегі көлденең кескіндерінен анықталды. Қабықшалардың қалыңдығы күйдіру ортасына байланысты өзгереді. NiO_x қабықшаларының EDX спектрлерін өлшеу жүргізілді. O/Ni сандық қатынасын талдау, ауа атмосферасында күйдірілген қабықша NiO стехиометриялық көрсеткіштеріне жақын екенін көрсетті. Комбинациялық шашырау спектрлері бойынша шыңдар қарқындылығының жоғарылауы байқалады, бұл күйдіру ортасына байланысты Ni бос орындарының қабықша ақауларының тығыздығының жоғарылауын көрсетеді. Жұтылу спектрлері алынды және NiO пленкаларының таук графиктері қабықшалардың оптикалық тығыздығы, қабықшаларды күйдіру жағдайларына байланысты. Деректерді талдау көрсеткендей, тыйым салынған аймақ ені күйдіру ортасына байланысты Eg = 3,12 эВ-ден Eg = 3,45 эВ-ге дейінгі аралықта. Кедергі спектрлерін талдау R_1 және R_2 кедергі мәндерін бағалауға мүмкіндік берді. R_1 кедергісінің жоғарылауы

NiO_x пленкасындағы ақаулардың тығыздығының төмендеуімен, R_2 рекомбинациялық кедергісінің төмендеуімен және рекомбинациялық процестердің жоғарылауымен байланысты екендігі көрсетілген.

Түйін сөздер: никель оксиді, золь-гель технологиясы, күйдіру ортасы, оптикалық тыйым салынған аймақ, жұтылу коэффициенті, дисперсия индексі, электр тасымалдау сипаттамалары.

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Эволюция структурных, оптических и электротранспортных свойств пленок NiO_x, отоженных в различных средах

В работе исследовано влияние среды отжига тонких пленок оксида никеля (NiO_x) на структурные, оптические и электротранспортные свойства. Пленки NiO_x были синтезированы золь-гель методом и отожжены при температуре 300 °C на воздухе, инертных газах азота (N_2), кислорода (O_2) и в вакууме. Значения толщины пленок определяли по поперечному сколу СЭМ-изображения. Толщина пленок зависит от среды отжига. Проведены измерения EDX-спектров пленок NiO_x. Анализ количественного соотношения O/Ni показал, что пленка, отожженная в атмосфере воздуха, близка к стехиометрическим параметрам NiO. Проведены измерения EDX-спектров пленок NiO_x. По спектрам КР наблюдается увеличение интенсивности пиков, свидетельствующее об увеличении плотности дефектов пленки вакансиями Ni в зависимости от среды отжига. Получены спектры поглощения и таук-графики пленок NiO_x.Оптическая плотность пленок зависит от условий отжига пленок. Анализ данных показывает, что ширина запрещенной зоны зависит от среды отжига и варьируется от Eg = 3,12 эВ до Eg = 3,45 эВ. Анализ спектров импеданса позволил оценить значения сопротивлений R_1 и R_2. Показано, что увеличение сопротивления R_1 связано с уменьшением плотности дефектов в пленке NiO_x, уменьшением рекомбинационного сопротивления R_2 и усилением рекомбинационных процессов на границе раздела.

Ключевые слова: оксид никеля, золь-гель технология, среда отжига, оптическая запрещенная зона, коэффициент поглощения, показатель дисперсии, характеристики электротранспорта.

Introduction

Among many conductive oxide materials, nanostructured nickel oxide (NiO_x) films attract considerable attention due to their unique structural, electrical and optical properties [1-2]. Numerous studies have made significant progress in the development and improvement of the properties of NiO_x, which has led to its widespread use in various electronic devices such as thin-film transistors, photovoltaic cells, LEDs, sensors, storage devices and catalysts [3].Nickel oxide is an inorganic semiconductor with p-type transport properties that can be used as a hole-conduction layer (HTL) material in optoelectronic devices [4]. NiO_x exhibits natural hole conductivity, as a rule, due to a large number of intrinsic defects, such as Ni vacancies and O vacancies. However, Ni vacancies are considered effective NiO_x acceptors, they also impair the transparency of the material in the visible area. It was found that the presence of a Ni vacancy in NiO_x grown in a medium with a high O content leads to significant absorption of subzones compared with an O vacancy in NiO_x grown in a medium with a high Ni content [5].

Various methods are used to improve the characteristics of NiO_x films, including changing the concentration of impurities, using various synthesis and post-treatment methods in UV ozone [6,7], oxygen plasma [8] and thermal annealing. The most common type of post-treatment is thermal annealing [9]. At the same time, the conditions of thermal annealing of the film are also of great importance. As a rule, in order to recrystallize the film and reduce the density of point defects, annealing is carried out in an oxygen-containing medium at a temperature of 200-500 °C and a duration of 10 to 60 minutes [10-13]. However, it is believed that atmospheric annealing does not allow the removal of NiOOH phases on the surface of an oxide semiconductor [14]. It is assumed that there is a high probability of the formation of NiOOH phases after heat treatment as a result of reaction with moisture from the atmosphere. The use of vacuum annealing can contribute to the thermal decomposition of the NiOOH phase. In addition, since the NiO_x phase is more stable than the Ni_2O_3 phase, at low oxygen partial pressure [15], vacuum heat treatment should reduce the content of this phase on the surface.

Thus, it is necessary to study technological methods for optimizing internal defects in NiO_x to simultaneously increase the conductivity and transparency of films, for the application of transparent optoelectronic devices.

In this work, NiO_x films were obtained by sol-gel method and the effect of thermal annealing in various media on the morphology, optical and electrical properties of NiO_x films was studied [16].

Materials and methods

Thin films preparation

Nickel oxide films were obtained on the surface of a solid substrate as follows: nickel nitrate hexahydrate [$Ni(NO_3)_2$ $6H_2O$] weighing m = 145 mg (Sigma-Aldrich) was dissolved in a volume of ethyleneglycol (V = 1 ml). Monoethanolamine (5μl) was added to the resulting solution. The solution was stirred at room temperature for 16 hours and then kept for 24 hours at room temperature. It was applied to the surface of a solid substrate using a pipetted is penser by centrifugation (SPIN150i, Semiconductor Production System) at a rotational speed of 1500 rpm. The obtained films were pre-annealed at a temperature of 100 °C for 15 minutes. After that, the films were annealed in different media at a temperature of 300 °C for 45 minutes (Fig. 1).

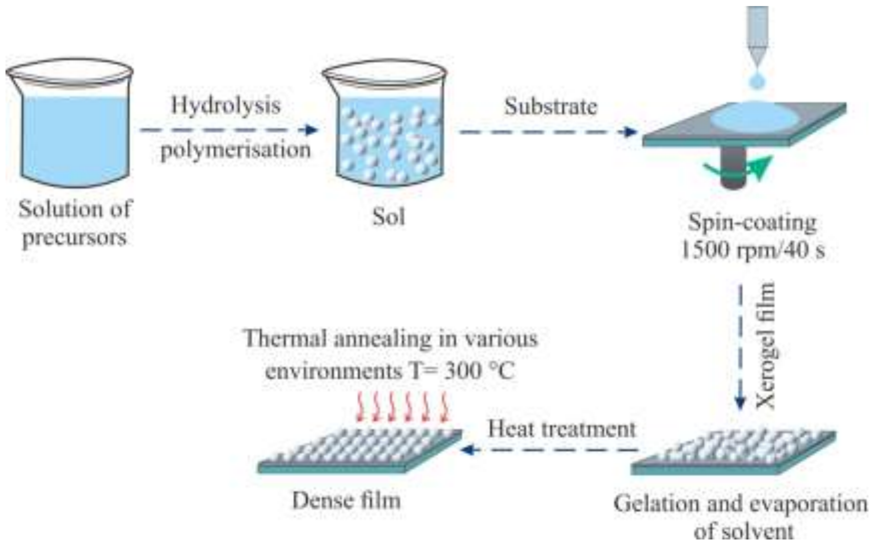


Figure 1 – Schematic representation of NiO_x thin films deposition from a sol by a spin-coating technique

The surface of NiO_x thin films was studied using scanning electron microscopy (SEM), MIRA 3 LMU (TESCAN). Energy dispersive X-ray radiation (EDX) is measured in an INCA PentaFET-x3 analyzer (Oxford Instruments, England) to assess the quality of thin films.

Scanning probe microscopy is used to study the surface roughness of NiO_x thin films using an atomic force microscope JSPM-5400 (AFM, JEOL). The AFM images were processed using a modular program for analyzing data from scanning probe microscopy (Win SPMII Data-Processing Software). The average grain size distribution of NiO_x was obtained from topographic images using ImageJ software.

The Raman spectra were measured using the Gurzil Raman Microscope TORMS-532/TORMS-785 (Thunder Optics, France). To excite the spectra, radiation from a semiconductor laser (ThunderOptics, France) with a wavelength of 523 nm and a line width of 0.3nm was applied.

The absorption and transmission spectra of the studied samples were measured on AvaSpec-ULS2048CL-EVO spectrometer (Avantes). A combined deuterium-halogen light source AvaLight-DHc (Avantes) with an operating range of 200-2500 nm was used as probing radiation.

The impedance spectra were measured on a P45X potentiostat-galvanostat in the impedance mode with an additionally installed FRA-24M frequency analyzer module. The error in determining the parameters of charge carrier transport did not exceed 5% and was predominantly 1-1.5%. Fitting and analysis of the spectral parameters were performed using the EIS-analyzer software according to the procedure described in [17].

Results and discussion

Surface morphology

Fig. 2 shows SEM images of the surface of nanostructured NiO_x films annealed at 300 °C in air, inert nitrogen (N_2), oxygen (O_2) and vacuum. As can be seen from the SEM image, the morphology of the NiO_x film in the annealed air atmosphere has a smoother surface structure. For films annealed in an inert N_2 atmosphere and in a vacuum, the surface morphology has a fine-grained structure. The film annealed in an O_2 atmosphere has a morphology with larger grains. The thicknesses of NiO_x films were determined from the SEM images of the transverse cleavage.It can be seen from the SEM image that the film annealed on an air atmosphere has the greatest thickness value of ~ 187 nm. For NiO_x films annealed in N_2, the thickness is ~ 89 nm. During annealing in O_2, a decrease in film thickness to ~ 76 nm is observed, the smallest film thickness of ~ 58 nm is obtained by annealing in vacuum.

Table 1 shows the results of EDX analysis of NiO_x films annealed at 300 °C in various media. For the EDX analysis, the NiO_x film was applied to Si substrates. According to the EDX spectra, a change in the quantitative ratio of the elemental composition of O/Ni is observed. Thus, when annealed in an air atmosphere, the NiO_x film becomes closer to the stoichiometric index (O/Ni ~ 0.8).

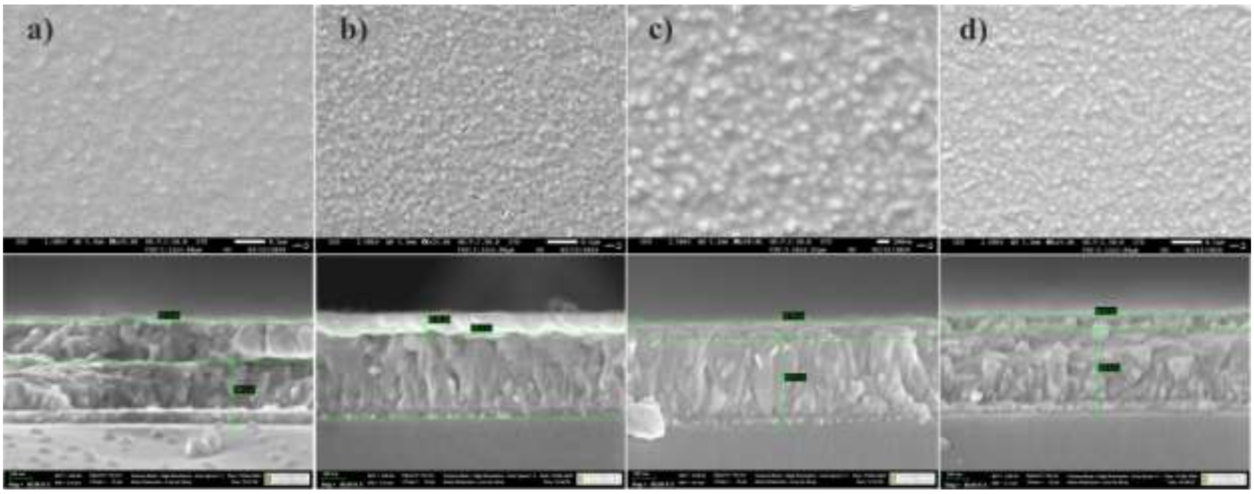


Figure 2 – Change in ambient temperature up to 300 °C in (a) air, (b) nitrogen, (c) oxygen and (d) vacuum

Table 1 – Morphology parameters and EDX analysis (Atomic percentages) of the surface of NiO_xfilms

Annealing ambient	Film thickness, Nm	Ni, %	O, %	Ni/ Oratio
Air	187	44.5	55.5	0.80
Nitrogen	89	33.4	66.6	0.50
Oxygen	76	18.0	82.0	0.21
Vacuum	58	42.3	64.4	0.65

Annealing of films in an inert atmosphere of N_2 and vacuum leads to a decrease in the stoichiometric index to 0.5. Annealing of films in pure O_2gas leads to a significant decrease in the atomic nickel content to 18% and the stoichiometric index by 0.21. Thus, quantitative EDX analysis of films annealed in inert N_2, O_2 gas and in vacuum indicates insufficient stoichiometry between Ni and O atoms.

Raman spectra of light in NiO_x films

Fig. 3 shows the Raman spectra of NiO_x films annealed in various media. The Raman spectra were measured in the range of 300 cm^{-1}–1500 cm^{-1}. In the raman light scattering spectra of NiO_x films, bands with a scattering maximum of 533 cm^{-1} corresponding to Ni-O oscillations belonging to a single-phonon longitudinal optical mode of the first order (1P) LO and a strong band with a scattering maximum of 1130 cm^{-1} relating to a two-phonon longitudinal optical mode of the second order (2P) 2LO are observed [18]. As can be seen from the Fig.3, the intensity of the bands depends on the annealing medium of the NiO_x films. The maximum scattering intensity is observed for NiO_x films deposited on the air atmosphere. The lowest scattering intensity has a NiO_x film pressed on an oxygen atmosphere. The observed increase in peak intensities in the scattering spectrum of films indicates an increase in the density of defects in the film formed by Ni vacancies depending on the annealing medium.

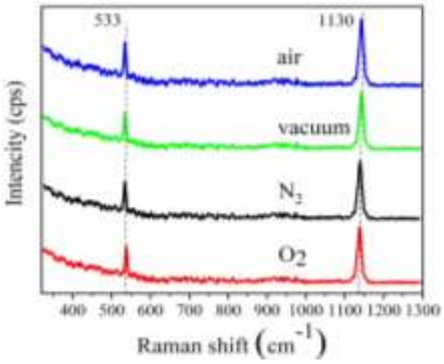


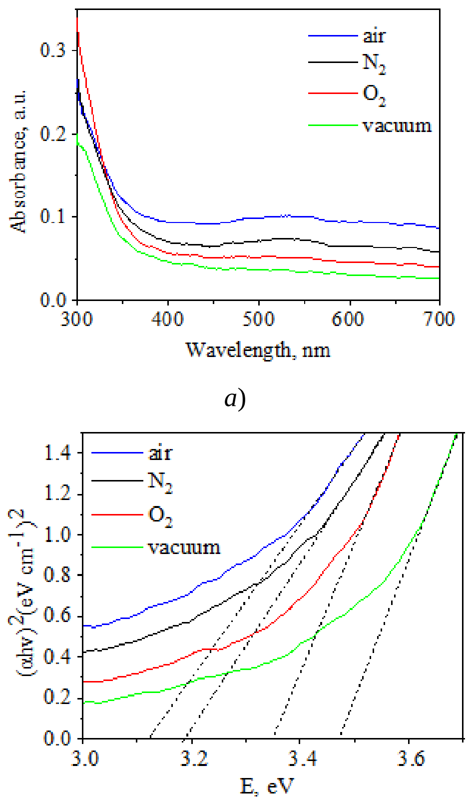
Figure 3 – Raman spectra for NiO_x films

Optical properties

Fig. 4 shows the absorption of NiO_x films obtained under various annealing conditions. It can be seen from Fig. 4a that the absorption spectrum of NiO_x has a maximum at a wavelength equal to $\lambda = 300$ nm. The absorption spectra of annealed films in air has an optical density of 0.25. During annealing in N_2, an increase in optical density to 0.26 is observed. The films annealed in O_2 have an increase in optical density up to 0.33. The optical density reduces to 0.19 for films annealing in vacuum. The absence of correlations between the thicknesses of films annealed in different media and the values of optical densities in the absorption spectra is associated with the compaction of the structure of NiO_x films, as shown in [19].

The observed good absorption of films in the short-wavelength region of the spectrum corresponds to the interband transition from the valence band to the bottom of the conduction band. This region corresponds to transitions from the final states of the valence band to the expanded states of the conduction band. A band in the long-wavelength part of the absorption spectrum of NiO_x films at $\lambda > 360$ nm is observed. The band may be associated with light scattering on grains in the NiO_x film. Fig. 4b shows the Tauc graphs for NiO_x films. The optical band gap was calculated by plotting the dependence $(\alpha h\nu)^2$ from photon energy E in eV (α is the absorption coefficient, $h\nu$ is the photon energy) and extrapolation of the rectilinear part of this graph to the energy axis.

Data analysis shows that the band gap width depends on the annealing medium and varies from $Eg = 3.12$ eV to $Eg = 3.45$ eV. This can be caused by both the high density of film defects and the high density of oxygen vacancies responsible for the high conductivity of films [20]. Interstitial oxygen in NiO_x films act as lattice defects and can reduce the optical band gap. In addition, interstitial oxygen atoms diffuse from NiO_x films during the thermal annealing process, as a result, the optical band gap Eg of NiO_x films increases.



a)

b)

Figure 4 – Absorption spectra (a) and Tauc plots (b) of NiO_x films obtained under various annealing conditions

Table 2 – Parameters of optical characteristics of NiO_x films under various annealing conditions

Annealing ambient	*D*, ($\lambda = 305$ nm)	Optical band gap (eV)
Air	0.20	3.12
Nitrogen	0.26	3.18
Oxygen	0.32	3.34
Vacuum	0.23	3.45

Photoelectrical characterizations

The influence of the annealing medium of NiO_x films will obviously affect the hole transport in NiO_x films. To study in detail the effect of the annealing medium on the transport of charge carriers in the NiO_x film, the electrical impedance spectra were measured.

Fig. 5 shows the impedance spectra of FTO/NiO_x/Al cells annealed in different media obtained using a hodograph fitting. The model was analyzed according to an equivalent electrical circuit using the EIS-analyzer software package(in Fig. 5). Table 3 shows the electrical transport parameters of the cells calculated, where R_1 is the total resistance of the external electrodes adjacent to NiO_x (total resistance of the film), R_2 is the recombination resistance at the NiO_x/electrode boundary.

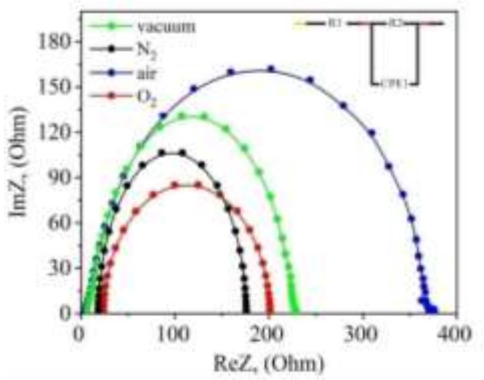


Figure 5 – Impedance spectra of FTO/NiO_x/Al, cells annealed in different environment

Table 3 – The value of the electrophysical parameters of the films

Annealing temperature, °C	R_1, (Ohm)	R_2, (Ohm)
Air	5.19	371.45
Vacuum	8.75	282.65
N_2	23.98	224.26
O_2	26.72	202.18

The analysis of the impedance spectra allowed us to estimate the values of the resistances R_1 and R_2. It is important to note that R_1 and R_2 depend on the bias voltage and cell configuration, however, if these parameters do not change, then changes in the values of R_1 and R_2 will be associated with changes in the structural properties of NiO_x during annealing in different media.

R_1 represents the total resistance of the outer adjacent layers (FTO/Al). In our case, all functional layers except NiO_x were obtained under the same conditions, then it is obvious that changes to R_1 will be associated with a change in the resistance of NiO_x films. As can be seen from Table 3, the value of R_1 has the lowest value for a cell with NiO_x annealed on an air atmosphere. The maximum resistance value R_1 has a NiO_x film annealed in an inert atmosphere N_2. An increase in the resistance value R_1 depending on the annealing medium is associated with a decrease in the density of defects formed by Ni vacancies depending on the annealing medium responsible for high conductivity [21].

The R_2 resistances characterizing charge recombination at the NiO_x/electrode interface also vary depending on the annealing medium of the NiO_x film. It should be noted that the higher the value of R_2, the lower the charge recombination rate at the NiO_x/electrode interface will be [22-25]. As can be seen from Table 3, cells with film annealed in the air atmosphere have the highest resistance value R_2, which indicates a low recombination rate of holes at the NiO_x/electrode interface. Annealing of NiO_x films in the O_2 atmosphere leads to a decrease in recombination resistance, which means an increase in recombination processes.

Conclusion

The structural, optical and electrical transport properties of NiO_x films produced by sol-gel technology were investigated. It was found from SEM images the surface of NiO_x films have a granular structure, the sizes of which depend on the annealing medium. According to EDX analysis of films, it was shown that films annealed in inert N_2, O_2 and in vacuum have insufficient stoichiometry between Ni and O atoms.The results of measuring the Raman spectra showed that the observed increase in peak intensities in the scattering spectrum of films is associated with an increase in the density of Ni defects depending on the annealing medium. It is shown that the band gap width depends on the annealing medium and is explained by the presence of interstitial oxygen in NiO_x films, which act as lattice defects, this leads to an increase in the optical band gap Eg. The values of the resistances R_1 and R_2 of NiO_x films annealed in different media estimated from the impedance spectra.It is shown that an increase in R_1 resistance depending on the annealing medium. It is associated with a decrease in the density of defects in the NiO_x film. Decrease in the recombination resistance (R_2) indicates an increase in recombination processes at the NiO_x/electrode interface. The results obtained will find applications of NiO_x thin films on electronic devices such as solar cells, gas sensors and catalysts cells.

Acknowledgments

This research is funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No.AP19679938).

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Article history:
Received 21 June 2024
Accepted 29 August 2024

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Мақала тарихы:
Түсті – 21.06.2024
Қабылданды –28.08.2024

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