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IMPROVING THE ELECTROPHYSICAL PROPERTIES OF NANOCOMPOSITE MATERIALS BASED ON GRAPHENE OXIDE AND TIO_2

This article shows the results related to the study of the effect of graphene oxide on the photoelectric properties of nanostructured titanium dioxide films. A TiO_2–GO nanocomposite material with a graphene oxide concentration of 3 wt% was synthesized by hydrothermal method. The Raman and IR spectra of nanocomposite materials have been studied and show the presence of peaks characteristic of graphene oxide and titanium dioxide. In the IR spectra, the so-called Ti-O-C bond is observed, which is responsible for fluctuations between TiO_2 and graphene oxide. Studies of absorption spectra show that in a nanocomposite material, the absorption spectrum is shifted to the long-wavelength region of light. Studies of electrophysical properties were carried out using electrochemical impedance spectroscopy. The impedance spectrum of this material shows that there is an improvement when graphene oxide is added to TiO_2 films. Photovoltaic parameters also show an increase in the photoinduced current in a nanocomposite material with the addition of graphene oxide by 3 wt%. It is shown that when graphene oxide is added 3 wt% to TiO_2, an increase in photocatalytic properties is observed due to an increase in the photoinduced current of the nanocomposite material.

Keywords: graphene oxide, titanium dioxide, TiO_2–GO, nanocomposite, photocatalysis, impedance spectrum, absorption spectrum.

Introduction

A brief overview of the materials. Among wide-band semiconductors, titanium dioxide occupies a special place because of its unique physicochemical properties and is widely used in photocatalysis, solar batteries, as well as in sensors [1–3]. Titanium dioxide belongs to the class of transition metal oxides. Titanium dioxide with anatase (tetragonal), brookite (rhombic), rutile (tetragonal) structures is often found in nature, titanium dioxide with a monoclinic TiO_2 (B) structure is rare, and TiO_2 (II) with the addition of a PbO_2 structure and TiO_2 (H) with a hollandite structure compound are also artificially synthesized [4-6].

Thanks to the development of nanotechnology, TiO_2 with various morphologies is already being synthesized: for example, nanotubes, nanorods, nanowires and mesoporous structures of titanium dioxide are already widely known and used [7]. The following methods are already used for the production and synthesis of TiO_2 nanoparticles: hydrothermal [8], solvathermal [9], sol-gel [10], chemical gas content (CVD) [11], sonochemical method [12], microwave [13], electrochemical oxidation of titanium [14], direct oxidation methods. Scientists also use new synthesis methods, for example, the supercritical fluid method [15], to synthesize nanoparticles with a high degree of homogeneity. The described methods can be used to produce not only TiO_2 nanoparticles, but also nanostructures with different morphology and modification, for example, nanorods, nanotubes, etc. The physicochemical properties of such films depend on the method of production and are determined by the particle size and shape, defect, phase composition, structure and pore sizes of the film.

Despite the similarity of the crystal structure of semiconductor nanostructures of titanium dioxide, the optical, photocatalytic, photovoltaic and electric transport properties are very different. Moreover, the changes may depend on many factors: on the process and method of production, the presence of impurities, geometry, morphology, specific surface area of nanostructures.

The variety of crystallographic forms of carbon puts this element in the center of attention both from the point of view of fundamental research and applied research. Three-dimensional crystallographic forms – graphite and diamond – have been known since ancient times and are widely used in industry. Relatively recently discovered zero-dimensional (fullerenes or cellular molecules) and one-dimensional (for example, carbon nanotubes) forms are now widely studied due to their remarkable and often unique mechanical and electronic properties.

Graphene, a planar, hexagonal arrangement of carbon atoms, has been the starting point in all calculations on graphite, carbon nanotubes and fullerenes since the late 1940s. However, his experimental discovery was postponed until 2004, when the micromechanical splitting method was used to obtain the first graphene crystals. The carbon atom has, accordingly, various modifications depending on the structure, for example – sp^3 hybridization is diamond, and its crystal lattice is in the form of a tetrahedral structure, sp^2 hybridization is graphite, fullerene and nanotubes are also in a tetrahedral structure, and sp hybridization is carbine in the form of a linear structure [16].

Graphene is a carbon layer in sp^2 hybridization consisting of condensed six–membered rings [17]. In other words, graphene is a single layer of graphite.

According to materials scientists, graphene (with a thickness of 1 layer) is a substance, not a material. And chemists say that graphene (also single–layer) is a polymer, while one molecule of this polymer-graphene will make up a mass of

only about one picogram. The graphene layer consists of carbon atoms located at a distance of 0.141 nm from each other in the nodes of the hexagonal lattice. In this case, each atom is connected to three neighboring atoms by covalent chemical bonds with sp^2 hybridization, and the fourth valence electron is included in the conjugated π-system of graphene. Thus, three bonds located in the plane define the geometric structure of graphene, and the fourth one defines its electronic properties [18].

The maximum charge mobility in a single-layer «suspended» graphene is 200,000 $cm^2/V*s$ [19]. This value is an order of magnitude (1-2) higher than the mobility of charge carriers in pure crystalline silicon, which implies using graphene as a rival for silicon in nanoelectronics.

The thermal conductivity of a graphene monolayer is 5000 W/m*K [20], this value is 10 times higher for copper and 2.5 times higher than for diamond. Thus, having high values of thermal conductivity, graphene can solve the problem of heat dissipation in nanoelectronics, where heating is a serious problem.

Gigantic strength. Graphene is the most durable material that has ever been measured. It is 100 times stronger than steel, harder than diamond. The strength of one layer corresponds to a Young's modulus of ~1.0 TPa. The stiffness coefficient for graphene exceeds the same index for diamond [21].

Elasticity. Graphene, despite its incredible strength, has flexibility and can undergo 20 % deformation without breaking the crystal lattice [22].

The permissible current density in graphene is six orders of magnitude higher than that for copper, which is explained by ballistic current flow, almost unrelated to heat generation [18].

The transparency of the monolayer is characterized by an optical transmission coefficient of ~ 97.7 %. The optical absorption of the thinnest matter in the universe is associated with the fine structure constant α: $\alpha \approx 1/137$. The absorption value of the monolayer (2.3 %) with a fine structure constant is related by the ratio: $2.3\% \approx \alpha/\pi$. The reason for this connection is that the current carriers in graphene behave as if they had no mass [18]. The excellent transmittance of graphene in a wide wavelength range makes it very attractive in optoelectronics as a transparent conductive coating.

Graphene was first obtained in 2004 by a unique method by K. Novoselov and A. Geim in a very simple way, which consisted in sequential mechanical splitting of highly ordered crystalline graphite to the thickness of one or more atomic layers [23].

The unique physical properties of graphene (high surface area, excellent conductivity, mechanical strength, transparency in the visible spectrum, etc.) make it very promising for use in various fields of science and technology, such as electronics, energy, biotechnology, etc. Along with graphene itself, its derivatives are of great interest: graphene oxide, reduced oxide graphene and graphene doped

with nitrogen. Theoretical and experimental studies show that graphene doping opens up new possibilities for the physics and chemistry of this unique material.

Currently, the most common method of obtaining graphene oxide is the Hammers method, as well as its various modifications [24].

Recently, the intensive development of technologies and industry graphene and its modifications have been the subject of various studies in which they were used to form composite materials with TiO_2 with improved photocatalytic characteristics.

This improvement is achieved by one or more of the following schemes:

1 – increases the surface area of TiO_2 due to its interaction with the two-dimensional matte structure of graphene and its derivatives;

2 – enhances the adsorption of aromatic pollutants due to their strong interactions with the aromatic network of graphene and its derivatives;

3 – reduces the recombination rate between positive holes and photogenerated electrons due to the electron conductivity of graphene and its derivatives, which act as an electron absorber for photogenerated electrons on the TiO_2 surface.

Over the past decade, papers have been published reporting the production of complexes based on graphene and TiO_2 nanoparticles [25–27]. The following methods are widely used for the synthesis of composites based on TiO_2 nanoparticles and graphene with its modifications: hydrothermal, solvothermal, mechanical mixing with/without ultrasound, sol-gel, methods of deposition of liquids, aerosols, UV irradiation, the method of reduction with hydrazine, chemical vapor deposition (CVD) and the method of centrifugation.

Thus, nanocomposite materials based on graphene oxide and TiO_2 are synthesized by various methods that show high adsorption capacity, high photodegradation of dyes, high efficiency of hydrogen decomposition. Each of the methods of nanocomposite synthesis has its own disadvantages and advantages, and some of them are even technologically difficult to synthesize particles.

One of the important properties of obtaining nanocomposite materials is the concentration of the initial components. The concentration of graphene oxide in TiO_2 strongly affects the electrophysical, photocatalytic and optical characteristics of the nanocomposite. There are many works using different concentrations in the synthesis of nanocomposites. Studies in this direction have shown that the optimal concentration is the ratio of 5 wt% graphene oxide to TiO_2 [28].

In this work, nanocomposite materials based on titanium dioxide and graphene oxide with a concentration of carbon-containing material equal to 3 wt% relative to TiO_2 were prepared.

Materials and methods

To prepare a 3 wt% nanocomposite based on TiO_2 and GO, the following was performed: 30 mg of GO was mixed with 90 ml of deionized water and 30 ml of

ethanol. Then the suspension was treated with ultrasound for 1 hour. After that, 1 g of TiO_2 was added and ultrasound and mixing were sequentially alternated for 2 hours, for each procedure for 30 minutes, until a homogeneous suspension of a uniform light gray color was achieved. The suspension was then placed in a Teflon-lined autoclave and left for 24 hours at 120 °C to synthesize the composite. After cooling to room temperature, the suspension was filtered several times with deionized water and ethanol. The resulting product is dried at a temperature of 60 °C.

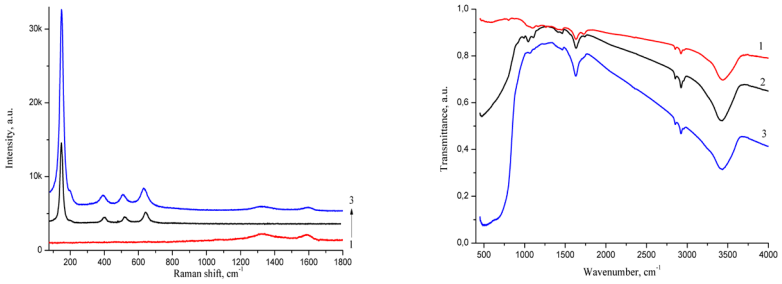
The finished paste was applied to the substrate surface by the «spin-coating» method and dried at a temperature of 100 °C for 30 minutes. The thickness of the film was controlled by the centrifuge rotation speed. After application and drying, the film was annealed in an argon atmosphere for 2 hours at a temperature of 450 °C.

Results and discussions

In order to confirm the reduction of graphene oxide during hydrothermal synthesis, Raman spectra of synthesized samples were recorded (Figure 1, a).

There are two characteristic bands in the spectrum of graphene oxide. The D-band of about 1350 cm^{-1} characterizes the degree of graphene defectiveness and is active only if the defects participate in double resonance scattering near the Brillouin zone [29]. The G-band is centered about 1590 cm^{-1}. And TiO_2 of anatase structure has six combinationally active peaks in the vibrational spectrum [30].

The Raman spectra of TiO_2-GO nanocomposites are a combination of the spectra of individual components. The ratio of the intensities of D- and G-bands in the nanocomposite material was calculated to be ID/IG equal to 1.2 for 3 wt%. The increase in ID/IG may be associated with an increase in bond defects or the formation of a large number of small sp^2-bound carbon domains during the reduction process [31,32].



a) b)

Figure 1 – Raman (a) and FT-IR (b) spectra:
1 – graphene oxide, 2 – TiO_2, 3 – TiO_2-GO/3 %

In the FT-IR spectrum (Figure 1, b) of graphene oxide, there are bands characterizing the fluctuations of oxygen-containing bonds: C–O (1095 cm^{-1}), C-O-C (1261 cm^{-1}), C-OH (1454 cm^{-1}), C=O (1728 cm^{-1}). An intense peak at 3441 cm^{-1} characterizes the fluctuations of OH groups in the composition of C-OH and water. The peak at 1628 cm^{-1} is associated with skeletal oscillations of graphene oxide [33]. The same bands were registered in the spectrum.

In a pure TiO_2 sample, there is a low-frequency mode of about 500 cm^{-1}, which corresponds to the vibration of Ti-O-Ti bonds. Also, as can be seen from the spectrum, an intense band appears around 3440 cm^{-1}, which indicates that OH groups are adsorbed on the surface of titanium dioxide particles.

The TiO_2–GO nanocomposite exhibits absorption below 1000 cm^{-1}. For the TiO_2-GO nanocomposite, this band is very pronounced, and its intensity increases with increasing GO concentration. This band is usually considered as a combination of bands corresponding to the fluctuations of Ti-O-Ti (695 cm^{-1}) and Ti-O-C (about 792 cm^{-1}) bonds [33]. This indicates that during the hydrothermal reaction, GO or rGO interacts through the residual functional groups of carboxylic acid with the surface hydroxyl groups of TiO_2 nanoparticles.

After the preparation of solid films of the triple nanocomposite material, the electrophysical characteristics of the TiO_2-GO nanocomposite material and pure TiO_2 were investigated. When studying the absorption spectra of nanocomposites, the data shown in Figure 2 were obtained. The absorption spectrum of TiO_2 is manifested in the UV region of the spectrum of about 380 nm. Graphene oxide also absorbs in the UV range, the maximum of its absorption spectrum is 230 nm. At the same time, GO films are practically transparent in the wavelength range from 400 to 800 nm [34,35].

When measuring the optical properties of the prepared films, it was found that the absorption band of the semiconductor broadens into the visible range of the spectrum in the nanocomposite material (Figure 2). It is also seen that the nanocomposite actively absorbs light in the UV region of the spectrum. Earlier in [36] it was shown that in nanocomposites there is a change in the band gap of the semiconductor. This contributes to a wider spectral sensitivity of nanocomposite materials, as well as to the improvement of their photoelectrochemical properties.

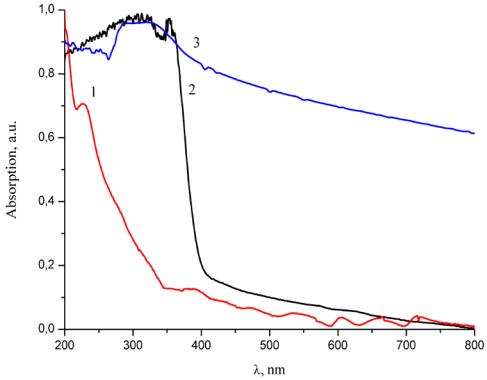
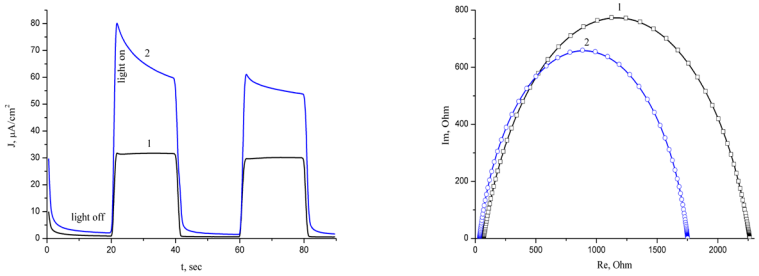


Figure 2 – Normalized absorption spectra of films:
1 – graphene oxide, 2 – TiO_2, 3 – TiO_2-GO/3 %

Further, the photoelectric characteristics of TiO_2 and TiO_2-GO/3 % were studied, with the help of which their photocatalytic activity can be estimated. The intensity of the photoinduced current was measured for 20 seconds with cyclic switching on and off of the light.

The magnitude of the photocurrent of a TiO_2-based film is ~30 μA (Figure 3a). When the sample was irradiated, the magnitude of the photocurrent increased by 2 times. Also, a high generation of photoinduced current for the first turn-on cycle was recorded for nanocomposite films.



a) b)

Figure 3 – Transient response of the photocurrent (a) and Impedance spectra in Nyquist plots (b) of samples: 1 – TiO_2, 2 – TiO_2-GO/3 %

Further, the electrophysical characteristics of the nanocomposite material were studied. Impedance spectra in Nyquist coordinates based on films are shown in Figure 3b. Based on the obtained impedance spectra, the main electric transport properties of the films were calculated. Parameters such as R_k, R_w, k_{eff}, and t_{eff} were determined, where R_k – charge-transfer resistance related to recombination of electron, R_w – electron transport resistance in TiO_2-GO, k_{eff} – effective rate constant for recombination and t_{eff} – effective lifetime of electrons [37].

Figure 3b shows that the diameter of the TiO_2 film hodograph is smaller than that of TiO_2 films. This means that the studied samples have a smaller amount of charge transfer resistance. The addition of graphene oxide makes it possible to reduce the resistance values R_k and R_w of semiconductor samples.

Table 1 shows the values of the electrophysical parameters of the TiO_2 film and the nanocomposite material. Using the EIS–analyzer software package, R_k and R_w are calculated, and k_{eff} is determined by the maximum of the hodograph arc using the formula $w_{max}=k_{eff}$. The thickness of the films was determined using a TESCAN Mira3 scanning electron microscope.

Таблица 1 – Electric transport parameters of TiO_2 and TiO_2-GO/3 %

Sample	k_{eff}, s^{-1}	τ_{eff}, ms	R_k, Ohm	R_W, Ohm
TiO_2	13.895	72	2194.0	69.3
TiO_2-GO/3 %	19.307	52	1716.3	34.9

In TiO_2, the charge transfer resistance is equal to R_k=2194.0 Ohms, and in TiO_2-GO/3 % is equal to R_k=1716.3.0 Ohms. Thus, the data show that when graphene oxide is added 3 wt%, the charge transfer resistance decreases by 20 %. It is also seen that the R_w – in the film is equal to 34.9 ohms, which means that the resistance to electronic transport is almost 2 times less than in TiO_2.

Conclusion

Samples based on TiO_2 and graphene oxide with the addition of 3 wt% were prepared and their optical, photoelectric and electrophysical characteristics were studied.

Measurements of the optical characteristics of the synthesized material showed that the absorption spectrum of the nanocomposite material corresponds to the spectra of the initial components. At the same time, there is a slight shift of the absorption band of the nanocomposite to the long-wavelength region. The transient characteristics of the photocurrents show an increase in the photoinduced current of the nanocomposite material. Studies of impedance spectra have shown that the addition of graphene oxide 3 wt% helps to reduce the resistance of semiconductor films.

The data obtained are an intermediate result in a study in which it was claimed that the best and optimal concentration of graphene oxide is 5 wt% relative to TiO_2.

The results obtained can be used to create photocatalytic materials for the UV and visible spectral ranges, as well as to be relevant in areas requiring photodegradation of organic compounds.

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ГРАФЕН ОКСИДІ МЕН TiO_2 НЕГІЗІНДЕГІ НАНОКОМПОЗИТТІК МАТЕРИАЛДАРДЫҢ ЭЛЕКТРОФИЗИКАЛЫҚ ҚАСИЕТТЕРІН ЖАҚСАРТУ

Бұл мақалада графен оксидінің наноқұрылымды титан диоксиді пленкаларының фотоэлектрлік қасиеттеріне әсерін зерттеуге байланысты нәтижелер келтірілген. Гидротермиялық әдіспен графен оксиді 3 мас% концентрациясы бар TiO_2–GO нанокомпозиттік материалы синтезделді. Графен оксиді мен титан диоксидіне тән шыңдардың болуын көрсететін нанокомпозиттік материалдардың Комбинациялық шашырау мен ИҚ-ның спектрлері зерттелінді. ІҚ спектрлерінде TiO_2 мен графен оксиді арасындағы тербелістерге жауап беретін Ti-O-C байланысы бар. Жұту спектрлерін зерттеу барысында нанокомпозиттік материалда жұту спектрі жарықтың ұзын толқын ұзындығына ауысқанын көрсетеді. Электрофизикалық қасиеттерді зерттеу электрохимиялық импеданс спектроскопиясы арқылы жүргізілді. Бұл материалдың импеданс спектрі TiO_2 қабыршақтарына графен оксидін қосқанда жақсару байқалатынын көрсетеді. Сонымен қатар, фотоэлектрлік параметрлер 3 мас% графен оксиді қосылған нанокомпозиттік материалында фотоиндукцияланған токтың жоғарылауын көрсетеді. Графен оксиді 3 мас% TiO_2-ге қосқанда нанокомпозиттік материалдың фотоиндукцияланған тогын ұлғайту арқылы фотокаталитикалық қасиеттердің жоғарылауы байқалады.

Кілтті сөздер: графен оксиді, титан диоксиді, TiO_2-GO, нанокомпозит, фотокатализ, кедергі спектрі, сіңіру спектрі.

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УЛУЧШЕНИЕ ЭЛЕКТРОФИЗИЧЕСКИХ СВОЙСТВ НАНОКОМПОЗИТНЫХ МАТЕРИАЛОВ НА ОСНОВЕ ОКСИДА ГРАФЕНА И TiO_2

В данной статье представлены результаты, связанные с изучением влияния оксида графена на фотоэлектрические свойства наноструктурированных пленок диоксида титана. Гидротермальным методом был синтезирован нанокомпозитный

материал TiO_2–GO с концентрацией оксида графена 3 мас.%. Были изучены спектры комбинационного рассеяния света и ИК-излучения нанокомпозитных материалов, которые показывают наличие пиков, характерных для оксида графена и диоксида титана. В ИК-спектрах наблюдается так называемая связь Ti-O-C, которая отвечает за колебания между TiO_2 и оксидом графена. Исследования спектров поглощения показывают, что в нанокомпозитном материале спектр поглощения смещен в длинноволновую область света. Исследования электрофизических свойств проводились с помощью электрохимической импедансной спектроскопии. Спектр импеданса этого материала показывает, что при добавлении оксида графена к пленкам TiO_2 наблюдается улучшение. Фотоэлектрические параметры также показывают увеличение фотоиндуцированного тока в нанокомпозитном материале с добавлением оксида графена на 3 мас.%. Показано, что при добавлении оксида графена 3 мас.% к TiO_2 наблюдается увеличение фотокаталитических свойств за счет увеличения фотоиндуцированного тока нанокомпозитного материала.

Ключевые слова: оксид графена, диоксид титана, TiO_2–GO, нанокомпозит, фотокатализ, импеданс спектр, спектр поглощения.

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