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PHOTOSENSITIVE STRUCTURES BASED ON THE SILVER-DOPED POROUS SILICON AND REDUCED GRAPHENE OXIDE

Igor Olenych , Lyubomyr Monastyrskii , Petro Parandiy ,
Roman Serkiz , Ivan Karbovnyk 
Ivan Franko National University of Lviv
50 Dragomanov St., Lviv 79005, Ukraine

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ABSTRACT

Background. The synergistic combination of useful properties of the different nature nanostructures in composites is one of the main methods of improving the functionality of nanomaterials. In particular, due to the unique properties of graphene as well as the large absorbing surface area and the low reflectance of porous silicon (PS), hybrid structures based on them are promising for photoelectric applications. Therefore, the possibility of increasing the photosensitivity of such structures by electrochemical introduction of silver nanoparticles into the porous layer was studied.

Materials and Methods. Nanostructured layers of the PS obtained by electrochemical etching of a silicon wafer and a film-forming suspension of reduced graphene oxide (RGO), which was deposited on the porous layer surface, were used to create photosensitive structures. The resulting structures with silver incorporated into the PS were explored using scanning electron microscopy. The electrical and photoelectric properties of the hybrid structures were studied based on the analysis of I - V characteristics and impedance frequency dependencies measured in the dark and under irradiation with white light.

Results and Discussion. It has been established that silver is deposited mainly in the surface layer of the PS and its amount depends on the duration of electrochemical introduction. Doping the porous layer with silver increases the electrical conductivity and capacitance of the PS–RGO structures. Irradiation of the surface of experimental structures with white light causes an increase in the forward and reverse current through the structures but has almost no effect on the lateral I - V characteristics due to shunting the RGO film by silver nanoparticles. Based on the analysis of impedance spectra in the 10^2 – 10^5 Hz frequency range, the effect of irradiation on the resistive-capacitive properties of hybrid structures was studied.

Conclusion. It was established that the incorporated silver forms additional current paths through the PS. The RGO film resistance is partially shunted by the silver nanoparticles as well. As a result, the obtained structures demonstrate an increase in conductivity both when the charge is transferred through the structure and along the surface. In addition, silver nanoparticles contribute to the accumulation of photogenerated charge carriers and their extraction from the PS.

Keywords: Porous silicon, reduced graphene oxide, silver nanoparticles, photosensitivity, impedance.



INTRODUCTION

An important area of R&D in nanoelectronics is improving the functionality of nanomaterials. The combination of useful properties of the different nature nanostructures in composite materials is one of the main ways to achieve this goal. Due to the synergistic effect, nanocomposites often demonstrate improved or completely new properties and therefore have a high potential for use in devices of various appointments. Special attention of researchers is focused on hybrid materials based on graphene, which have the prospect of application in solar cells, energy storage devices, sensors, medicine and the food industry [1-4].

The outstanding properties of graphene are due to the conical shape of the electronic spectrum of the hexagonal structure of a monolayer of sp^2 -bonded carbon atoms [5, 6]. As a result, 2D graphene is characterized by high charge carrier mobility, high thermal conductivity, and the ability to withstand significant current density, which is a significant advantage for creating high-speed and powerful electronic devices [7-9]. The low electrical noise of the carbon monolayer provides a high sensitivity of graphene-based nanocomposites to the local electric field of adsorbed molecules [10]. Besides, graphene can be used as a transparent electrode in photoelectric devices due to its light absorption of only 2.3 % [11]. However, the high transparency of graphene and the high recombination rate of photoinduced charge carriers cause the weak photoresponse of this nanomaterial. One of the main strategies for improving the photosensitivity of carbon nanostructures is hybridization: the combination of graphene and light-sensitive semiconductors [12, 13]. In particular, hybrid structures based on reduced graphene oxide (RGO) film and porous silicon (PS) are promising for photovoltaic applications [14] because the PS layers are characterized by a large absorbing surface area and excellent anti-reflective properties [15, 16]. However, the high resistance of the PS imposes some limitations on the application of the material in photoelectronics, which requires finding ways to increase the efficiency of charge transfer through the porous layer.

This study aimed to explore the influence of electrochemical doping of the porous layer with silver on the electrical and photoelectric properties of the hybrid structures based on the PS and RGO film.

MATERIALS AND METHODS

The PS layer for photosensitive hybrid structures was obtained by electrochemical etching of a 400 μm thick silicon wafer of electronic conductivity type with a 4.5 $\text{Ohm}\cdot\text{cm}$ resistivity. An ethanol solution of hydrofluoric acid with the component ratio of $\text{HF}:\text{C}_2\text{O}_5\text{OH} = 1:1$ was used as the electrolyte. The anodic current density and etching time were 30 mA/cm^2 and 10 min, respectively. The working surface of the silicon wafer was irradiated with a 500 W lamp to increase the etching efficiency. A thin gold film that served as an electrical contact was previously deposited on the silicon substrate back surface by thermal technique and annealed at a 600 $^\circ\text{C}$ temperature for 20 min.

The doping of the PS layer with silver was carried out from an aqueous solution of silver nitrate (AgNO_3) using the electrochemical method. The value of the direct current was 1 mA for 5, 10 and 20 s to obtain different experimental samples. The resulting structures are labeled as sample 1, sample 2, and sample 3, respectively. In the case of passing the electric current through the electrolyte, metal ions are transferred to the cathode and deposited not only on the surface but also in the pores of the PS. Silver ions, which have a more positive electrochemical potential than silicon, after depositing on the surface of the walls between the pores are neutralized by withdrawing electrons from surface silicon atoms and become the nuclei for the growth of silver nanocrystals. In addition, redox processes can contribute to the formation of an oxide film on the PS surface. The incorporation of silver into the porous layer was controlled using scanning electron

microscopy (SEM) in the elastically reflected electron mode and energy-dispersive X-ray microanalysis (EDXMA).

After washing the formed structures with distilled water, a film-forming RGO suspension obtained by chemical reduction of graphene oxide as described in [17] was deposited on the PS layer surface. Further air-drying of the experimental samples at room temperature ensured the formation of the RGO film with a thickness of up to several tens of nanometers [14, 17]. Two silver contacts were thermally deposited on the RGO film surface at a 1 mm distance from each other.

The electrical and photoelectric properties of the obtained structures based on the Ag-doped PS and RGO film were studied in DC and AC modes using the Siglent SDM 3045 multimeter and the Hantek 1833C RLC-measuring device. Photoelectric phenomena were investigated under the condition of irradiation of the experimental structures from the RGO film side with a white light LED FYLP-1W-UWB-A with a 1 W power and a luminous flux of 76 lumens. All the measurements were carried out at room temperature

RESULTS AND DISCUSSION

The morphology of hybrid structures based on the Ag-doped PS and RGO film was studied using SEM in the secondary electron mode. As a result of electrochemical etching, the formation of long narrow pores directed into the depth of the silicon wafer perpendicular to the surface was observed (Fig. 1). The pore diameter ranged from tens to hundreds of nanometers, which facilitated the effective penetration of the electrolyte into the pores during the silver deposition process. The walls between the pores were also characterized by a nanometer thickness.

In the elastically reflected electron mode, the image areas with higher brightness are identified as formed silver clusters. Analysis of SEM images of the cross-section of the experimental structures made it possible to establish that a significant amount of

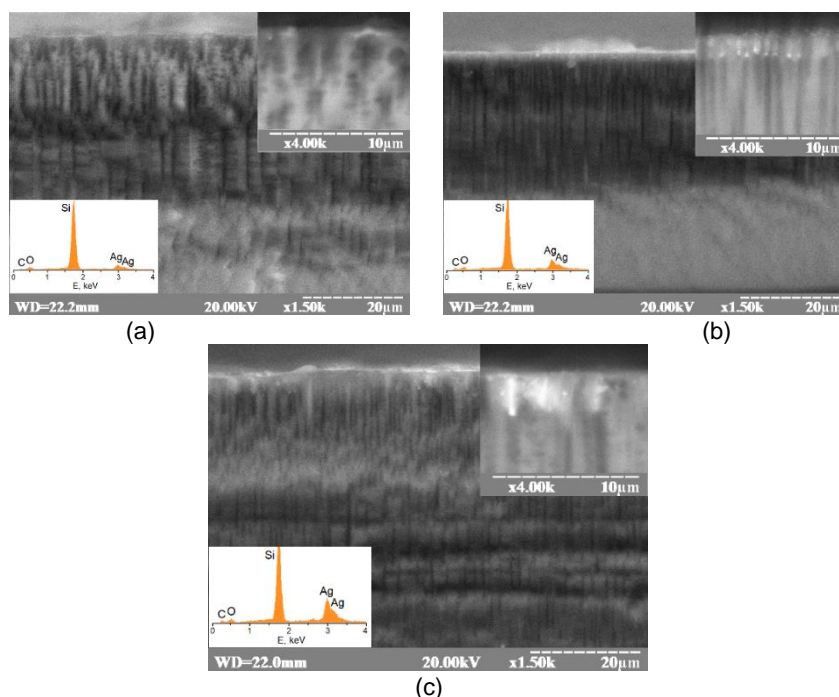


Fig. 1. SEM images of cross-sections of structures based on the Ag-doped PS and RGO in the secondary electron mode: sample 1 (a), sample 2 (b) and sample 3 (c). The insets show SEM images in the elastically reflected electron mode and EDXMA spectra of the corresponding structures.

electrodeposited silver was located in the near-surface layer of the PS. The surface of the walls between the pores in the upper part of the PS layer is well decorated with metal particles. An increase in the amount of incorporated silver is observed with an increase in the time of its electrochemical introduction into the porous layer. An intense maximum with energy of 1.74 keV associated with silicon atoms was detected in the EDXMA spectra. In addition, peaks at 2.98 and 3.15 keV that correspond to silver atoms were identified. The intensity of these peaks was greater for samples with higher amounts of metal. Small bands with energies of 0.3 and 0.5 keV are related to carbon and oxygen atoms, respectively, and can be associated with RGO and the native oxide film on the surface of the PS nanostructures.

Doping the porous layer with silver significantly affects the charge transfer processes in hybrid structures based on the PS and RGO both along the surface of the structures and in the direction perpendicular to the surface. In the first case, the main influence on charge transfer is the electrical properties of the RGO film because the resistance of the PS layer significantly exceeds the resistance of graphene.

As can be seen in Fig. 2, the lateral $I_{||}$ - $V_{||}$ characteristics are linear at small bias voltages. It is worth noting that the $I_{||}$ - $V_{||}$ characteristics are determined not only by charge carriers injected into the RGO film from electrical contacts but also from the PS layer and silver nanoparticles. An increase in the amount of incorporated silver causes a decrease in lateral resistance several times, which may be due to the formation of percolation clusters of metal nanoparticles that partially shunt the RGO film. A further increase in the duration of the silver electrochemical introduction probably causes the formation of additional current paths along the structure surface. As a result, a three-order decrease in the resistance between the contacts on the RGO film surface is observed (see the inset of Fig. 2).

The I_{\perp} - V_{\perp} characteristics of the Ag-doped PS-RGO structures have a nonlinear nature in the case when the current passes perpendicular to the surface (Fig. 3). An increase in the amount of silver incorporated into the porous layer causes increasing the forward current, which corresponds to a positive potential on the RGO film. The duration of doping the PS with silver has almost no effect on the magnitude of the reverse current in the dark. The observed shape of the I_{\perp} - V_{\perp} curves indicates the presence of several electrical barriers in the studied structures. Considering the Ohmic contact of graphene with metals, the dominant barrier is likely to be at the interface of the PS with silver nanoparticles that connect with the RGO film.

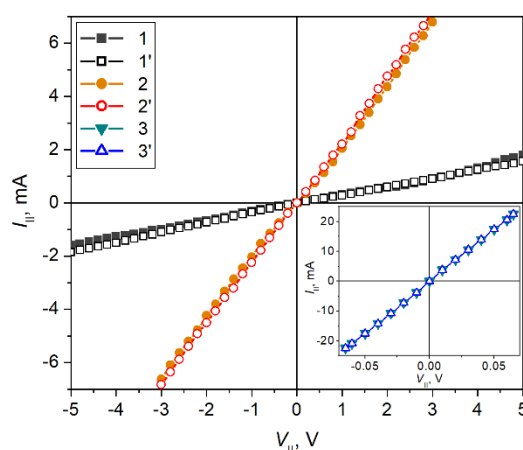


Fig 2. Lateral $I_{||}$ - $V_{||}$ characteristics of the Ag-doped PS-RGO structures obtained in the dark (curves 1, 2 and 3) and under irradiation with white light (curves 1', 2' and 3'). Curves (1, 1'), (2, 2') and (3, 3') related to samples 1, 2 and 3, respectively.

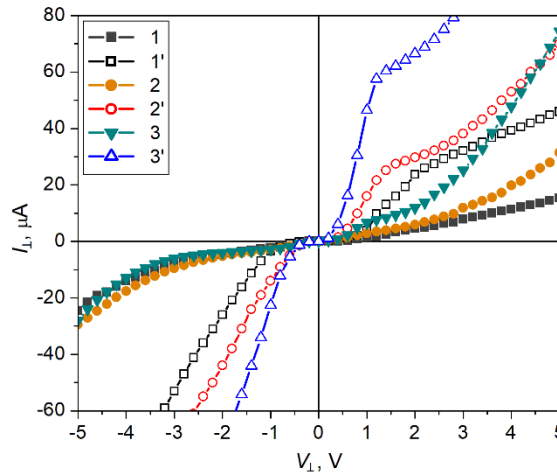


Fig 3. The I_L - V_L characteristics of the Ag-doped PS-RGO structures obtained in the dark (curves 1, 2 and 3) and under irradiation with white light (curves 1', 2' and 3'). Curves (1, 1'), (2, 2') and (3, 3') related to samples 1, 2 and 3, respectively.

An increase in both the forward and reverse current I_L is observed due to photogenerated charge carriers when the Ag-doped PS-RGO structures are irradiated with white light. Experimental samples with a higher amount of incorporated silver demonstrate higher photocurrent values. The incorporation of silver provides an increase in the efficiency of photoelectric structures based on the PS and RGO due to the high electron work function and a larger Schottky barrier area. In addition, silver nanoparticles can better collect light-induced charge carriers and extract them from the porous layer. In the forward current mode, the maximum influence of photocarriers on the I_L - V_L curves is observed at the small bias voltage (up to 1.2–2.0 V for various samples), after which the shape of the current-voltage dependencies becomes similar to ones in the dark. Identified features of the I_L - V_L characteristics may be an additional argument in favor of the assumption of the participation of incorporated silver in the processes of accumulation of photogenerated charge carriers.

It is worth noting that the lateral III-VII characteristics of hybrid structures almost do not change when irradiated with the white light LED (see Fig. 2). This fact may be due to the high transparency of the RGO film and the low-quality dielectric properties of the silver-doped PS layer if the studied structures are used as graphene field-effect transistors. Moreover, silver nanoparticles not only shunt the RGO film but can also block the local electric field of charge carriers that are photogenerated in the PS layer and the silicon substrate.

The frequency dependences of the impedance between the RGO film and the silicon substrate were measured to study the capacitive-resistive properties of the Ag-doped PS-RGO structures. A decrease in capacitance and internal resistance with increasing frequency was observed in the 10^2 – 10^5 Hz range (Fig. 4).

The different dispersion of the electrical parameters of the experimental samples in various frequency ranges along with the identified features of the I_L - V_L characteristics indicate complex processes of charge transfer through the Ag-doped PS-RGO structure perpendicular to the surface. Usually, low-frequency and high-frequency parts of impedance dependence are associated with the transfer of charge through the interfaces and in the bulk of nanostructures, respectively. Nyquist plots were used to analyze frequency dependencies of the impedance of the experimental structures (Fig. 5).

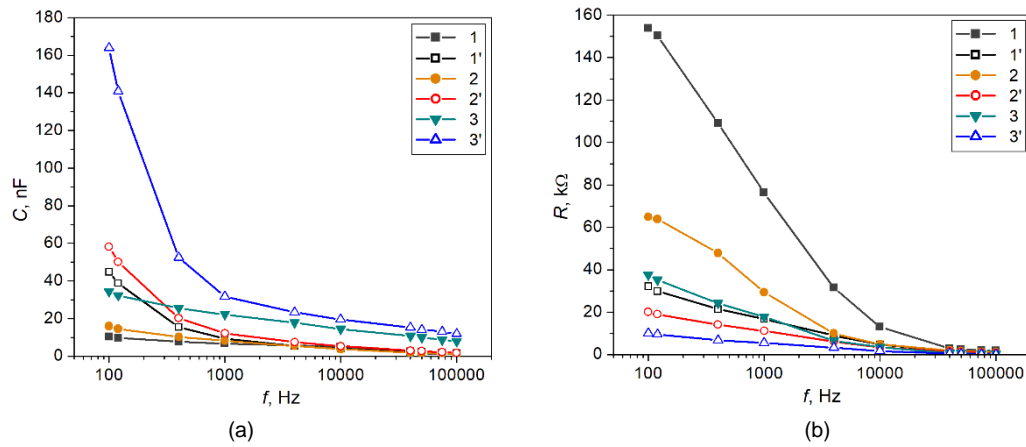


Fig. 4. Frequency dependencies of capacitance (a) and internal resistance (b) of the Ag-doped PS-RGO structures obtained in the dark (curves 1, 2 and 3) and under irradiation with white light (curves 1', 2' and 3'). Curves (1, 1'), (2, 2') and (3, 3') related to samples 1, 2 and 3, respectively.

A parallel-connected constant phase element (CPE) and resistor with the resistance R were applied in the equivalent circuit model considering the capacitive-resistive properties of fractal and composite materials [14, 18]. The impedance of the R–CPE model is defined by the equation

$$Z(\omega) = \frac{R}{1 + (j\omega)^n RQ}. \quad (1)$$

Here Q is the CPE and n characterizes the heterogeneity of the electrical properties of the Ag-doped PS–RGO structures. The parameters of approximation of the impedance spectra in the 400 Hz – 100 kHz frequency range are shown in Table 1.

Based on the analysis of the approximation results, it can be concluded that the capacitive-resistive properties of the PS–RGO hybrid structures depend on the amount of incorporated silver in the porous layer. Increasing the duration of the doping process

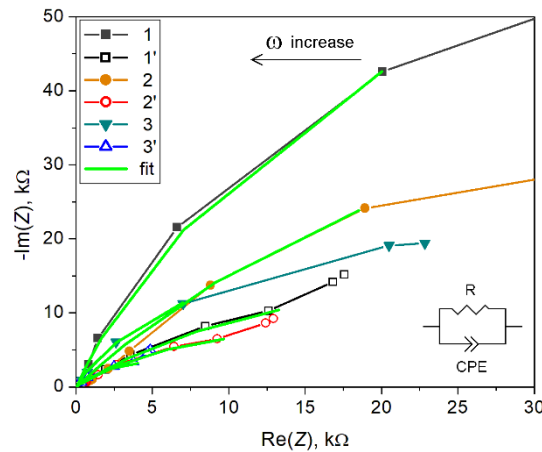


Fig 5. Nyquist plots and an equivalent circuit diagram of the Ag-doped PS–RGO structures obtained in the dark (curves 1, 2 and 3) and under irradiation with white light (curves 1', 2' and 3'). Curves (1, 1'), (2, 2') and (3, 3') related to samples 1, 2 and 3, respectively.

Table 1. The parameters of approximation of the impedance spectra of the Ag-doped PS–RGO structures

Ag-doped PS–RGO structures	Parameters of approximation					
	In the dark			Under irradiation		
	R , k Ω	$Q \times 10^{-7}$	n	R , k Ω	$Q \times 10^{-7}$	n
Sample 1	206.64	0.23	0.86	51.59	4.34	0.59
Sample 2	139.75	1.18	0.70	28.19	5.67	0.59
Sample 3	44.34	0.99	0.83	13.08	6.70	0.68

causes an increase in the conductivity and capacitance of the studied structures due to the formation of additional current paths through the PS layer and a larger area of the Schottky barrier.

Besides, the Ag-doped PS–RGO structures demonstrate an increase in capacitance and a decrease in internal resistance under white light illumination from the LED FYLP–1W–UWB–A.

CONCLUSION

In the work, photosensitive structures were created by depositing a film-forming suspension of RGO onto the PS surface and further air-drying at room temperature. An incorporation of silver nanoparticles into the porous layer was proposed to increase the efficiency of photovoltaic structures. Using SEM methods, it was demonstrated that silver is deposited mainly in the surface layer of the PS and its amount depends on the duration of electrochemical introduction.

Based on the measurement of the I–V characteristics, it was found that the incorporated silver forms additional current paths. As a result, the obtained structures demonstrate an increase in electrical conductivity both when the charge is transferred through the structure and along the surface. An increase in forward and reverse current through the Ag-doped PS–RGO structure was detected under the influence of white light irradiation. However, illumination of the hybrid structure surface has almost no effect on the lateral I–V characteristics due to the RGO film shunting and blocking of the local electrical field of photogenerated charges by silver nanoparticles. An increase in capacitance and a decrease in internal resistance between the RGO film and the silicon substrate both due to increasing the duration of electrochemical incorporation of silver and white light exposure have been established based on the analysis of the impedance frequency dependencies in the 10^2 – 10^5 Hz range. The obtained results can be used to create photodetectors based on hybrid nanomaterials.

COMPLIANCE WITH ETHICAL STANDARDS

The authors declare that they have no competing interests.

AUTHOR CONTRIBUTIONS

Conceptualization, [I.O., L.M., P.P.]; methodology, [I.O., L.M., P.P.]; investigation, [I.O., P.P., R.S., I.K.]; writing – original draft preparation, [I.O.]; writing – review and editing, [I.O., L.M., P.P.]; visualization, [I.O., R.S., I.K.].

All authors have read and agreed to the published version of the manuscript.

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ФОТОЧУТЛИВІ СТРУКТУРИ НА ОСНОВІ ЛЕГОВАНОГО СРІБЛОМ ПОРУВАТОГО КРЕМНІЮ ТА ВІДНОВЛЕНОГО ОКСИДУ ГРАФЕНУ

**Ігор Оленич, Любомир Монастирський, Петро Парандій,
Роман Серкіз, Іван Карбовник**

*Львівський національний університет імені Івана Франка,
вул. Драгоманова 50, 79005 м. Львів, Україна*

АНОТАЦІЯ

Вступ. Синергетичне поєднання корисних властивостей наноструктур різної природи у композитах є одним із основних методів підвищення функціональності наноматеріалів. Зокрема, завдяки унікальним властивостям графену, великій площі поглинаючої поверхні та низькому коефіцієнту відбивання поруватого кремнію гібридні структури на їх основі є перспективними для фотоелектричних застосувань. Тому у роботі вивчена можливість підвищення фоточутливості таких структур шляхом електрохімічного впровадження у поруватий шар наночастинок срібла.

Матеріали та методи. Для створення фоточутливих структур використано наноструктуровані шари поруватого кремнію, одержаного електрохімічним травленням кремнієвої пластини, та плівкоутворювальну суспензію відновленого оксиду графену, яку осаджували на поверхню поруватого шару. Отримані структури з інкорпорованим у поруватий шар сріблом досліджено методами скануючої електронної мікроскопії. Електричні та фотоелектричні властивості гібридних структур досліджено на основі аналізу вольт-амперних характеристик і частотних залежностей імпедансу, виміряних в темноті та при освітленні білим світлом.

Результати. Встановлено, що срібло переважно осаджується у приповерхневому шарі поруватого кремнію, а його кількість залежить від тривалості електрохімічного впровадження. Легування поруватого шару сріблом зумовлює збільшення електропровідності та ємності структур на основі поруватого кремнію та відновленого оксиду графену. Опромінення поверхні експериментальних структур білим світлом спричиняє збільшення прямого і зворотного струму через структури, але майже не впливає на латеральні вольт-амперні характеристики через шунтування плівки відновленого оксиду графену наночастинами срібла. На основі аналізу спектрів імпедансу у частотному діапазоні 10^2 – 10^5 Гц досліджено вплив опромінення на резистивно-ємнісні властивості гібридних структур.

Висновки. Встановлено, що інкорпороване срібло утворює додаткові шляхи проходження струму через поруватий кремній і частково шунтує плівку відновленого оксиду графену. Як наслідок, отримані структури на основі поруватого кремнію та відновленого оксиду графену демонструють збільшення електропровідності при

перенесенні заряду як через структуру, так і вздовж поверхні. Крім того, наночастинки срібла сприяють накопиченню фотогенерованих носіїв заряду та їх виведенню з поруватого шару.

Ключові слова: Поруватий кремній, відновлений оксид графену, наночастинки срібла, фоточутливість, імпеданс.