

Hydrogen Treatment of a Plasmon Resonance Sensor

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The effect of hydrogen on a surface plasmon resonance sensor was studied. The sensor is a glass plate of 1 mm thickness with a gold film applied to one surface. To increase the adhesion of the gold film on the glass a thin chromium interlayer between the gold film and the glass plate was applied. Thickness of the chromium layer was 5 nm. The thickness of the gold film was 50 nm. Hydrogen treatment was performed by means of electrolysis in a 10 % water solution of sulfuric acid (H_2SO_4). The surface of the gold film served as the cathode. Electrolysis lasted for 6 min. At room temperature due to electrolysis, the gold film was saturated with water. During the hydrogen treatment of the sensor, constant electric currents were maintained. A series of samples were treated with different electrolysis currents (50-2 μA). The influence of the proton flux density to the surface of the gold film on the properties of the surface plasmon resonance sensor was studied. For this purpose, the proton flux density during hydrogen treatment was changed from $8.86 \cdot 10^{13}$ to $3.47 \cdot 10^{12}$ $1/(cm^2 \cdot s)$. Optical properties of treated and untreated sensors were studied experimentally by means of Plasmon-5 spectrometer. Hydrogen treatment changed the optical properties of the surface plasmon resonance sensor over time after hydrogen treatment. The biggest changes occurred during the first days, which gradually slowed down. It was established that after hydrogen treatment the surface plasmon resonance curve was shifted in the direction of larger angles in comparison with the case of an untreated sensor. It was also found that treatment with large fluxes of protons is less effective in changing the optical properties of the sensor than with smaller ones.

Keywords: Electrolysis, Hydrogen treatment, Hydrogen diffusion, Surface plasmon resonance.

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1. INTRODUCTION

Hydrogen has unique physical properties. It is the first element of the periodic table. Therefore, the smallest size of hydrogen atom allows to "settle" it in the interatomic space of the crystal lattice of many materials. Dissolving in materials, it loses a single electron and turns into a very mobile proton particle in the crystal. Hydrogen is able to pass into the metal during melting, casting and various chemical, electrochemical, gas discharge and nuclear processes. Numerous studies are devoted to the material saturation. Hydrogen can damage the material. For example, hydrogen saturation can lead to a decrease in the properties of plasticity (hydrogen fragility), the formation of cavities, cracks and other structural imperfections, which facilitate rapid destruction of constructive elements in contact with hydrogen [1]. In other cases, the penetration of hydrogen into the material leads to transformations that change properties of the material in the desired direction. Namely, hydrogen is used to process materials. Hydrogen treatment of materials allows to create unique physical properties of materials [2-4].

Free-state hydrogen is almost absent in nature, but it is easily obtained by contact of hydrogen-containing compounds with a metal surface. Contact of the metal surface with water is a fertile medium for the gradual dissolution of hydrogen in the metal. The water contains $6 \cdot 10^{13}$ protons in one cubic centimeter at room temperature. In an acidic solution, the number of H^+ ions per cubic centimeter is greater than indicated.

Surface plasmon is a surface electromagnetic wave that occurs at the boundary of media with high conductivity (metals, semiconductors) and dielectrics (air or water solutions). Surface plasmons are resonance fluctuations in the density of free charge carriers in the near-surface layer of a conductor or semiconductor that occur under the action of electromagnetic radiation. Excitations of charge density create oscillations of an electromagnetic field of considerable amplitude. The oscillations of the electromagnetic field are localized near the boundary and rapidly weaken on both sides. The amplitude of the field strength fluctuations can be strong enough to help break the chemical bonds of hydrogen in the molecules. That is, under the action of plasmons there is an additional dissociation of water molecules or the rupture of hydrogen bonds with molecules in colloidal solutions.

Devices that use surface plasmon resonance, due to high sensitivity of the method, are used in many modern studies of microbiology, chemistry and medicine [5-7]. The surface plasmon resonance sensor usually contains a required element. As a rule, that is a thin film of gold on the surface of the dielectric. Such sensors are used in devices "Plasmon-5" and "Plasmon-6", which were developed and manufactured at the V. Lashkaryov Institute of Semiconductor Physics NAS of Ukraine.

The main aim of the present paper is to study changes of optical properties of a treated by hydrogen nanoscale gold film deposited on the surface of the glass film with chromium interlayer between the gold film and the glass plate.

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2. EXPERIMENTAL STUDIES

2.1 The Samples for Research

Gold films of 50 nm thickness thermally deposited on glass plates were used as the samples for research. In the present study, the samples with dimensions of 20.0×20.0 mm were used. In the thermal deposition method, gold has low adhesion with glass. Therefore, to improve adhesion of the gold film, a layer of chromium of 5 nm thickness was deposited on the glass plate before deposition of the gold film.

The technological cycle of sample production starts from preparation of substrate, which are glass plates of 1 mm thick. On this stage, the glass plates were subjected to chemical treatment in an ultrasonic bath, washed in deionized water in an ultrasonic bath, dried and annealed in an oven at 200 °C for 20 min. Then, the vacuum chamber was pumped. The substrates were loaded into cassettes. Immediately after that, cassettes with the substrates were installed into the working vacuum chamber for the deposition of the chromium layer and the gold film.

A 50 nm thick gold film was deposited on the activated silicon surface and glass substrates. To obtain gold films, the method of direct contact heating in a tungsten shuttle (energy was transferred to phonons) in an industrial unit of VUP-5M at a residual gas pressure in the working chamber of 10^{-3} Pa was used. The deposition was performed on a substrate at room temperature. The deposition rate of gold was 0.5 nm/s.

2.2 Hydrogen Treatment of the Samples

Hydrogen treatment of the samples was performed by means of electrolysis. The electrolytic bath was filled with a 10 % water solution of sulfuric acid (H_2SO_4). The gold film was connected to the negative pole of the current source for electrolysis through a gold-plated metal contact. The side surface of the metal contact attached to the film was much smaller than the surface of the film itself. Therefore, when calculating the hydrogen flux density to the film, the lateral surface area of the metal contact can be neglected. The positive pole of the current source was connected to a graphite electrode. Thus, during electrolysis, the gold film was the cathode and the graphite electrode was the anode. Electrolytic saturation of hydrogen occurred at room temperature. During electrolysis, direct current was maintained and video surveillance was conducted.

Hydrogen was collected near the surface between the gold film and the glass after diffusion through the entire thickness of the gold film. Current fluctuations did not exceed 5 %. The electrolysis process lasted for 6 min. At a current of 50 μA at the end of the first minute, bubbles with hydrogen were observed on the surface of the gold film. At a current of 5 μA , the formation of hydrogen bubbles on the surface of the gold film was not observed during the entire electrolysis period. Simple calculations revealed the number of ions reaching a single surface of the sample in contact with the electrolyte every second (proton flux density) and throughout the electrolysis time. Calculations were performed for all current values and results are shown in Table 1. After electrolysis, the samples were washed with distilled

water. Next, the samples were dried under running air at room temperature and stored at room temperature. As one can see from Table 1, in experiments the effect of changes in the flow of protons on the surface of the film was studied.

Table 1 – The proton flux density made by electrolysis

N_0	$I, \mu A$	Proton flux density, $1/(cm^2 \cdot s)$	Protons for all time, $1/cm^2$
1	50	$8.68 \cdot 10^{13}$	$5.21 \cdot 10^{16}$
2	5	$8.68 \cdot 10^{12}$	$5.21 \cdot 10^{15}$
3	2	$3.47 \cdot 10^{12}$	$2.08 \cdot 10^{15}$

2.3 Surface Plasmon Resonance Changes after Hydrogen Treatment of Gold Film

All studies of plasmon resonance were performed by the use of the PLASMON-5 installation. This is a computer-controlled spectrometer, which excites plasmons by the Kretschmann method. The device has a goniometer that changes the angle of incidence of the light beam incident to the surface of the gold film. Surface plasmons are excited on the surface of the gold film at the gold-air boundary by means of polarized light emitted by a semiconductor laser with a wavelength of 650 nm. Under the appropriate conditions of plasmon resonance, surface plasmons are excited. The excitation of plasmons is evidenced by a resonant decrease in the intensity of reflected light. The angular dependence of the intensity of the reflected beam of light is shown by the resonance curve of the surface plasmon resonance (SPR). Depending on the modification of the device, it has one or two optical channels. We used a device with two optical channels. Angular scanning took place within 10° . The accuracy of setting the angle of incidence was 10 angular seconds. SPR curves were obtained before and after hydrogen treatment of the samples. For comparison, these curves are given in the following figures.

SPR measurements were performed at various time intervals after hydrogen treatment. In the first hours after treatment, it was observed that regardless of the proton flux density during processing, all SPR curves change their angular positions over time (after hydrogen treatment) non-monotonically. For example, consider the details that took place in the case of a sample treated with hydrogen with a flow of protons of $8.6 \cdot 10^{13} 1/(cm^2 \cdot s)$ (Fig. 1).

From Fig. 1a, one can see that in the first hour after hydrogen treatment, the SPR curve shifted to larger angles and this shift was 0.20° . During the next hour, there was a shift of the SPR curve to smaller angles. The SPR curve almost returned to the position fixed before the hydrogen treatment. Three hours after hydrogen treatment, the maximum SPR angle changed by 0.11° was greater in the SPR curve before hydrogen treatment. Subsequently, with increasing time (exposure of the sample at room conditions) after hydrogen treatment, the maximum angle of the SPR curve increased. Twenty-four hours after hydrogen treatment, the angle of maximum of the SPR curve relative to the maximum before treatment increased by 0.16° , i.e. almost corresponded to the angle observed in the first hour after hydrogen treatment.

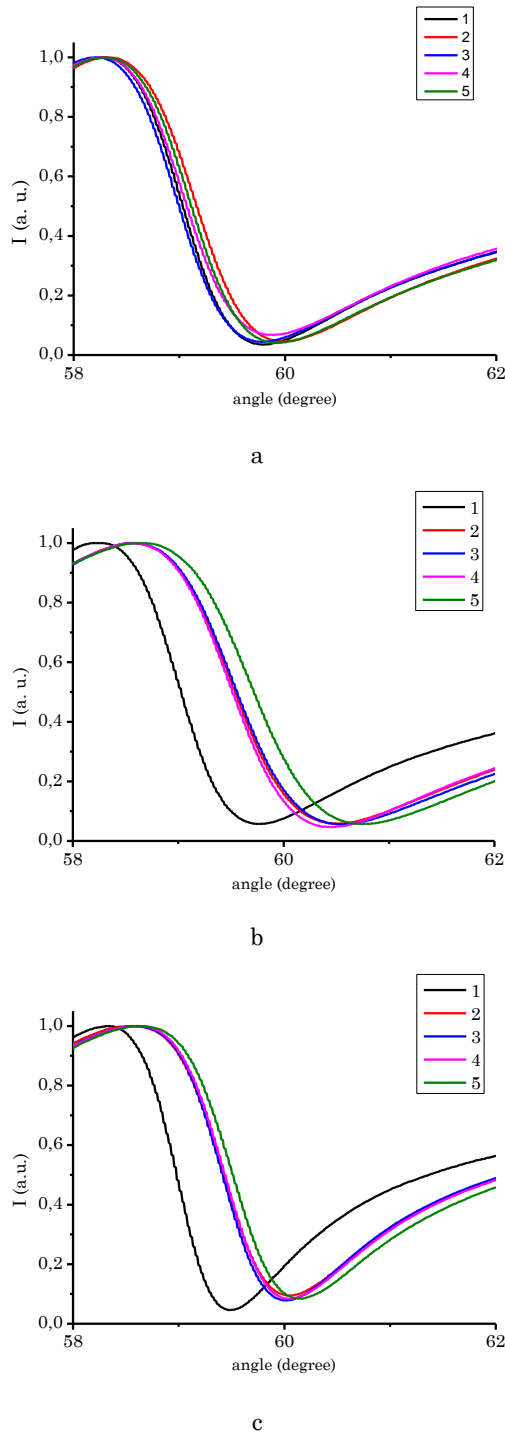


Fig. 1 – The SPR curves for samples treated with hydrogen with a proton flux density to the surface: a – $8.6 \cdot 10^{13} \text{ 1}/(\text{cm}^2 \cdot \text{s})$; b – $8.6 \cdot 10^{12} \text{ 1}/(\text{cm}^2 \cdot \text{s})$; c – $3.47 \cdot 10^{12} \text{ 1}/(\text{cm}^2 \cdot \text{s})$. SPR curves: 1 – before hydrogen treatment and after hydrogen treatment at time intervals: 2 – 1 h, 3 – 2 h, 4 – 3 h, 5 – 24 h

Let us follow the shift of the SPR curve during hydrogen treatment (in the first 24 h of exposure at room conditions) with a proton flux of $8.6 \cdot 10^{12} \text{ 1}/(\text{cm}^2 \cdot \text{s})$ (Fig. 1b). In the first hour after hydrogen treatment, the maximum of the SPR curve (relatively to the curve before treatment) shifted by 0.65° . During the next hour, this shift increased slightly and amounted to 0.69° . Three hours after hydrogen treatment, the small-

est shift of the maximum angle of the SPR curve of 0.59° was recorded. Twenty-four hours after hydrogen treatment, the maximum of the SPR curve shifted to larger angles, exceeded the maximum shift in the first hour and was 0.83° .

Let us follow the motion of the SPR curve during hydrogen treatment (in the first 24 h of exposure at room conditions) with a proton flux of $3.47 \cdot 10^{12} \text{ 1}/(\text{cm}^2 \cdot \text{s})$ (Fig. 1c). In the first hour after hydrogen treatment, the maximum of the SPR curve (in comparison with the curve before treatment) shifted by 0.57° . In the next hour, this shift did not change. Three hours after hydrogen treatment, the shift of the maximum angle of the SPR curve of 0.59° was recorded. Twenty-four hours after hydrogen treatment, the maximum of the SPR curve shifted to larger angles, exceeded the shift obtained in the previous measurements and was 0.68° .

Let us make some generalizations based on the results of the study. At higher currents in the first hour after hydrogen treatment, the shift of the maximum angle of the SPR curve is smaller at the largest flow of protons studied. In the following hours, this shift of the maximum angle of the SPR curve decreases, the SPR curve shifts to smaller angles than it was after the first hour. The SPR curves corresponding to the proton flux densities of $8.6 \cdot 10^{12} \text{ 1}/(\text{cm}^2 \cdot \text{s})$ and $3.47 \cdot 10^{12} \text{ 1}/(\text{cm}^2 \cdot \text{s})$ are characterized by a slight decrease in the angle of the SPR maximum after the first hour. After hydrogen treatment, all samples were washed in distilled water and dried at room temperature. It is known that after contact of water with the surface of a solid body, a nanolayer of water molecules remains on the surface for some time. It was decided to check how the SPR curve would change after contact with distilled water. Fig. 2 shows the SPR curves before contact with water and after at different times after contact.

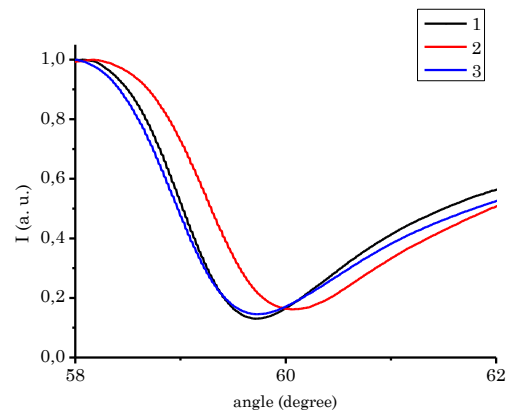


Fig. 2 – The SPR curves: 1 – before contact and after contact with water at time intervals: 2 – 1 h, 3 – 4 h

Fig. 2 shows that in the first hour after "drying" of water at room conditions, a nanolayer of water molecules remained on the surface of the gold film. The shift of the SPR curve relative to the curve before contact with water was 0.37° . Four hours after contact of water with the gold film, the maxima of the SPR curves before contact and after contact with water coincided, but the SPR curve corresponding to the sample after contact became slightly wider. Incomplete coincidence of

the SPR curves may be due, for example, to the presence of a small amount of solute in distilled water. After evaporation of water molecules, the molecules that were in solution remained. What conclusion can we draw from this observation? In the first hours after hydrogen treatment in the changes of the SPR curves

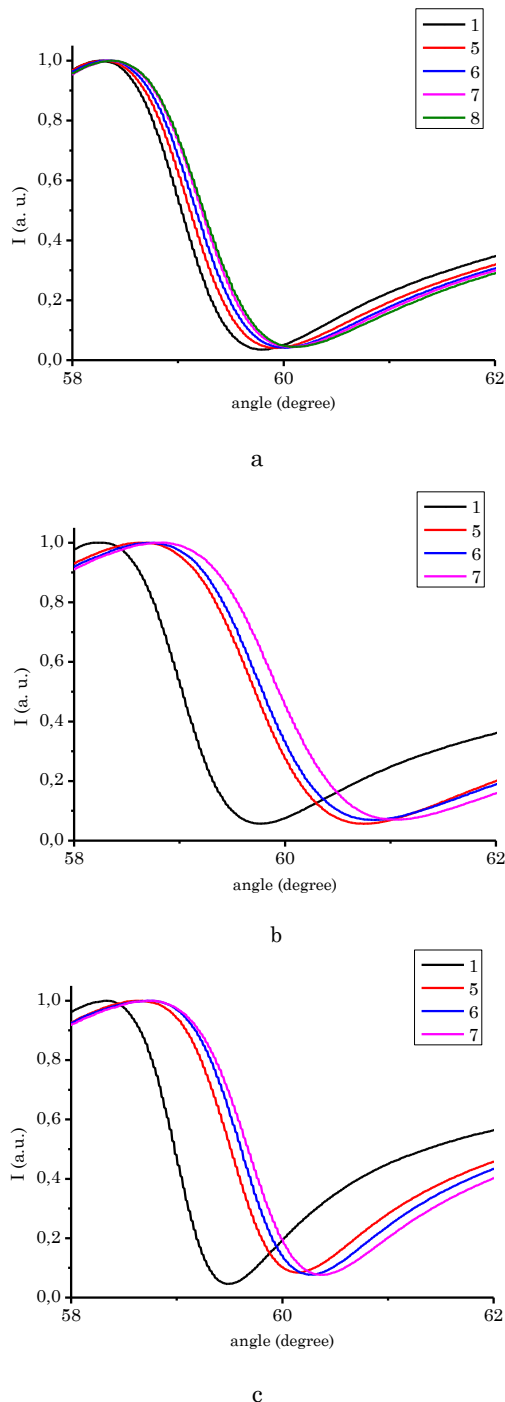


Fig. 3 – The SPR curves for samples treated with hydrogen with a proton flux density: a – $8.68 \cdot 10^{13}$ $1/(\text{cm}^2 \cdot \text{s})$ and 1 – before hydrogen treatment and after hydrogen treatment at time intervals: 5 – 24 h, 6 – 72 h, 7 – 144 h, 8 – 336 h; b – $8.6 \cdot 10^{12}$ $1/(\text{cm}^2 \cdot \text{s})$ and 1 – before hydrogen treatment and after hydrogen treatment at time intervals: 5 – 24 h, 6 – 168 h, 7 – 336 h; c – $3.47 \cdot 10^{12}$ $1/(\text{cm}^2 \cdot \text{s})$ and 1 – before hydrogen treatment and after hydrogen treatment at time intervals: 5 – 24 h, 6 – 168 h, 7 – 480 h

there is a contribution of water residues on the surface of the gold film. The results of hydrogen treatment of the gold film on the glass can be evaluated starting from 24 h after hydrogen treatment.

Consider the motion of the SPR curve for a sample treated with a proton flux of $8.68 \cdot 10^{13}$ $1/(\text{cm}^2 \cdot \text{s})$ on the following days after treatment (Fig. 3a). Over time, the SPR curve gradually shifted to larger angles relative to the curve obtained before sample processing. In the first days of observation, the rate of a relative shift was greater. During the last 7 days of observation, the SPR curve shifted to larger angles relative to the previous observation by only 0.02° . In general, during the whole observation period (14 days) after treatment, the SPR curve shifted by 0.29° .

Consider the motion of the SPR curve for a sample treated with a proton flux of $8.68 \cdot 10^{12}$ $1/(\text{cm}^2 \cdot \text{s})$ on the following days after treatment (Fig. 3b). Over time, the SPR curve gradually shifted to larger angles relative to the curve obtained before sample processing. In the first days of observation, the rate of a relative shift was greater. So from the first to the seventh day the average shift occurred from 0.05 angular degrees per day. Over the next 7 days, the SPR curve shifted averagely by 0.036° per day. In general, during the entire observation period (14 days) after treatment, the SPR curve shifted by 1.33° .

Consider the motion of the SPR curve for a sample treated with a proton flux of $3.47 \cdot 10^{12}$ $1/(\text{cm}^2 \cdot \text{s})$ on the following days after treatment (Fig. 3c). Over time, the SPR curve gradually shifted to larger angles relative to the curve obtained before sample processing. In the first days of observation, the rate of a relative shift was also higher. So from the first to the seventh day the average shift occurred from 0.025 angular degrees per day. Over the next 13 days, the SPR curve shifted by an average of 0.005° per day. In general, for the entire period of observation (20 days) after treatment, the SPR curve shifted by 0.89° .

3. DISCUSSION OF RESULTS

In the case of hydrogen treatment used at room temperature, the process of diffusion of hydrogen atoms through the film is the slowest. That is, the rate of hydrogen saturation of the film is controlled by the diffusion of hydrogen atoms through the gold film. Taking into account the size of the sample it is possible to suppose that we deal with a one-dimensional diffusion problem. The initial concentration of hydrogen in the film should be considered equal to zero everywhere except the surface in contact with the electrolyte. A constant concentration is maintained on the surface of the gold film. The problem of diffusion through the gold film with such initial and boundary conditions has already been solved, for example, in [8]. Evaluation of the diffusion through the gold film using this solution shows that at the boundary of the glass film, the hydrogen concentration one minute after the start of saturation will not differ by more than one percent from the concentration on the surface in contact with the electrolyte. This means that in such conditions, hydrogen saturates the gold film to the maximum possible concentrations in one minute.

Because the gold film is in contact with the glass, this connection has many defects. Defects are known to be good hydrogen traps [9, 10]. In these defects, hydrogen forms molecules and gradually accumulates. The pressure of the molecules gradually increases and this leads to the growth of pores. Table 1 (the last column) shows the number of hydrogen atoms that take part in electrolysis. Obviously, during electrolysis a part of hydrogen atoms remains on the surface of the film and bubbles are created, which are released into the atmosphere.

After the electrolysis process, the concentration of hydrogen on the surface of the film will decrease to zero. The process of hydrogen diffusion in the opposite direction will begin. The hydrogen atoms in the inter-nodes of the crystal lattice will leave the sample in a few minutes. Atoms that have been in vacancies at the nodes of the crystal lattice will come out of the film 10-100 times longer. That is, after a few hours the presence of hydrogen in these defects should be neglected. Hydrogen in the pores on the gold-glass interface forms molecules. Note that chemisorption is required to degas this hydrogen. At room temperature, chemisorption is almost impossible. That is, hydrogen in the pores will be stored indefinitely.

The right question arises. What is the relation of hydrogen bubbles formed at the gold-glass interface to the changes in SPR, since the gold-air interface is sensitive to changes in the SPR? Changes occurring at the gold-glass interface have little effect on SPR. Consider a model of processes that may occur in the case of hydrogen treatment of gold film. These processes will help to explain Fig. 4.

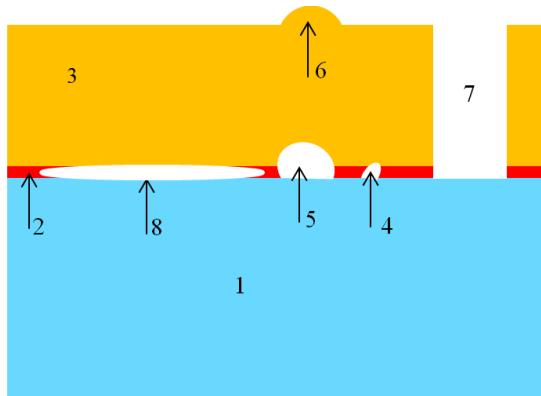


Fig. 4 – Damage to the film by hydrogen treatment: 1 – glass plate, 2 – chromium layer, 3 – gold film, 4 – small cavities with hydrogen, 5 – cavities with hydrogen of medium size, 6 – convexity of the surface of the gold film on the gold-air interface, 7 – holes in the gold film, 8 – large cavities with hydrogen

Consider the possible damage to the film that occurs after hydrogen treatment (Fig. 4). In the first case, small cavities contain hydrogen under high pressure. But small cavities do not change the relief of the gold-air interface. In the second case, cavities of medium size and with rather high inner hydrogen pressure are created. Under the pressure in cavities, the gold film is stretched and convexities of the surface are formed at the air-gold interface. In the third case, there are cavities of medium size with hydrogen pressure exceeding the strength of gold. In such cavities, a part of the film breaks off and holes are formed in the film. In the fourth case, there are

cavities of medium size (area on the gold-glass interface) with low hydrogen pressure, which does not change the relief of the gold-air interface. In the last case, large cavities with low hydrogen pressure do not change the relief of the air-gold interface.

With the flow of protons during electrolysis at $8.68 \cdot 10^{13} \text{ 1/(cm}^2 \cdot \text{s)}$, large cavities are most likely to form at the gold-glass interface. Cavities of medium size are also created. A part of them (with high hydrogen pressure) creates convexities. Other ones (that exceed the strength of gold) create holes. Small cavities are also created, but their number is the smallest. After electrolysis, the gold film is in a stress state, which gradually decreases. Due to the high pressure in the small cavities, they move, unite with each other and the larger cavities. Due to the small number of small cavities, it is possible to explain the insignificant increase in the shift of the SPR curve in the case of treatment with such a flow of protons during electrolysis. The flow of $8.68 \cdot 10^{12} \text{ 1/(cm}^2 \cdot \text{s)}$ is ten times less than $8.68 \cdot 10^{13} \text{ 1/(cm}^2 \cdot \text{s)}$. That is, ten times less hydrogen during electrolysis will reach the gold-glass interface. Hydrogen is not enough to form "scaly" pores, but enough to form pores with noticeable convexity and small pores with high inner pressure of hydrogen. This explains the significant change in plasmon resonance in the first day after hydrogen treatment and on the following. The flow of $3.47 \cdot 10^{12} \text{ 1/(cm}^2 \cdot \text{s)}$ is less. However, in such a case collected hydrogen is still enough to excite the gold-air interface, both on the first day after electrolysis and later in time.

Obviously, for describing the phenomenon of hydrogen treatment effect on plasmon resonance the hypothesis was discussed here above. To model the processes accurately, additional studies of the film surface are required. It will allow to visually examine the changes. However, the search for methods of formation of convexities on the surface of the gold film is important because such formations will increase the sensitivity of the plasmon resonance method. Importantly, in the present studies it was found the interval of proton flux from $1 \cdot 10^{13}$ to $5 \cdot 10^{12} \text{ 1/(cm}^2 \cdot \text{s)}$ in which one can observe an effective influence of hydrogen treatment on the optical properties of the gold film. Changes in the properties of the film lead to a significant shift of the SPR profile and a slight change in the magnitude of the resonance maximum.

4 CONCLUSIONS

Experimental study of the effect of hydrogen on the optical properties of the gold film on the glass, which is the sensor of the spectrometers "Plasmon 5" and "Plasmon 6", allowed to establish:

1. At hydrogen processing of the surface of the film by a flux of protons less than $8.68 \cdot 10^{13} \text{ 1/(cm}^2 \cdot \text{s)}$ there are changes of an angle of SPR. The amplitude of the SPR peak does not decrease. Changes in the resonance of plasmons are observed over time. These changes fade within 20 days.

2. The largest shift of the SPR profile to larger angles was recorded for a sample treated with a proton flux of $8.68 \cdot 10^{12} \text{ 1/(cm}^2 \cdot \text{s)}$, this shift was 1.33° .

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Обробка воднем плазмонного резонансного датчика

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Досліджено вплив водню на параметри датчика поверхневого плазмонного резонансу. Датчиком слугує скляна пластинка товщиною 1 мм з нанесеною на одну поверхню золотою плівкою. Для підвищення адгезії золотої плівки на скло було нанесено тонкий шар хрому між золотою плівкою і скляною пластинкою. Товщина шару хрому складала 5 нм. Товщина золотої плівки становила 50 нм. Воднева обробка була виконана за допомогою електролізу у 10 % водному розчині сірчаної кислоти (H₂SO₄). Поверхня золотої плівки слугувала катодом. Електроліз тривав 6 хв. При кімнатній температурі, завдяки електролізу, золота плівка була насичена воднем. Під час водневої обробки датчика підтримували постійні електричні струми. Була оброблена серія зразків різними струмами електролізу (50-2 μA). Досліджено вплив щільності потоку протонів до поверхні золотої плівки на властивості датчика поверхневого плазмонного резонансу. З цієї метою щільність протонного потоку під час обробки воднем змінювали з 8,86·10¹³ до 3,47·10¹² 1/(см²·с). Оптичні властивості оброблених і необроблених сенсорів були досліджені експериментально за допомогою спектрометра Plasmon-5. Воднева обробка змінила оптичні властивості датчика поверхневого плазмонного резонансу протягом часу після водневої обробки. Найбільші зміни відбувались протягом перших діб і поступово уповільнювались. Встановлено, що після водневої обробки крива поверхневого плазмонного резонансу змістилася у бік більших кутів відносно кривої, отриманої до обробки воднем. Установлено, що обробка більшими потоками протонів менш ефективно впливає на зміну оптичних властивостей датчика, ніж меншими.

Ключові слова: Електроліз, Обробка воднем, Дифузія водню, Поверхневий плазмонний резонанс.