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## OPTICAL AND PHOTOLUMINESCENT PROPERTIES OF GOLD NANOSTRUCTURES OBTAINED BY PULSED LASER DEPOSITION IN VACUUM

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The method of pulsed laser deposition in vacuum is used for the formation of gold films and composite films of aluminum and silicon oxides with gold nanoparticles. We measured the transmission spectra, as well as the time-resolved photoluminescence spectra in the visible spectral region, and established the conditions for the formation of gold nanostructures, whose extinction spectra include a resonance band related to the manifestation of local surface plasmons. A high-intensity photoluminescence with a band maximum at 2.2–2.4 eV and relaxation times reaching 5–6  $\mu\text{s}$  was for the first time observed for  $\text{Al}_2\text{O}_3$  films with gold nanoparticles obtained at the gold concentration in the target less than 20% and having no plasmon absorption band. The relation between the optical and photoluminescent properties of films and the nature of photoluminescence connected with the interband electron-hole recombination in gold nanoparticles are discussed.

### 1. Introduction

Metal nanoparticles (NP) demonstrate new optical and photoluminescent properties that differ from those of a bulk material. Their investigation allows one to develop conceptions about the rules governing the variation of their properties under the change of dimensions from the atomic (via those of clusters and nanocrystals) to bulk ones. On the other hand, metal (in particular, gold and silver) NPs are in demand for the improvement of characteristics of the existing device structures, as well as for the development of new ones. A peculiarity of the optical properties of noble metal nanostructures consists in the presence of resonance absorption bands related to local surface plasmons – collective oscillations of conduction electrons. Optical spectra of Au NPs depend on their dimensions, form, and dielec-

tric surrounding, which provides a possibility to control the optical properties. In order to investigate the local surface plasmon resonance (LSPR) of gold nanostructures, one used free clusters obtained in various ways, island (granulated) films, films containing Au NPs in oxide matrices, and rough films with a developed microrelief [1–9].

Optically excited metal surfaces demonstrate either weak or no photoluminescence (PL). In 1969, Mooradian [10] for the first time observed PL at the wavelength  $\lambda = 520 \text{ nm}$  with a quantum efficiency equal to  $10^{-10}$  for smooth films of bulk gold. During a long time, one could not raise the PL intensity even for rough gold films. But the situation has abruptly changed in the 1990s – the beginning of the 21st century when the subject of investigation became gold nanoparticles. The PL quantum efficiency in Au nanocolumns and nanoclusters smaller than 15 nm in size has increased by five-six orders of magnitude. The position of PL peaks covered a wide range of wavelengths from the visible to near infrared spectral regions. As a rule, PL relaxation times did not go beyond the nanosecond range. Fluorescence was observed more often [11–21].

The investigation of PL in noble metals has started from the already mentioned work [10]. The nature of PL in gold was associated with the radiative recombination of electrons of the  $6sp$  conduction band below the Fermi level and holes of the  $5d$ -band. In the phenomenological model of PL proposed by Boyd and coauthors [11], this radiative recombination was intensified by local fields caused by oscillations of surface plasmons of gold. In [14,19], PL was explained by the processes of

radiationless recombination of excited holes of the 5d-band and 6sp-electrons accompanied by the emission of surface plasmons that subsequently radiated. PL mechanisms of Au NPs are still discussed.

Photoluminescent properties are characteristic of various gold nanostructures obtained in different ways. Among them, there are island gold films formed by means of nanostructuring [14], Au:oxide composite multilayer systems, where Au NPs are located between SiO<sub>2</sub>, ZnO, or TiO<sub>2</sub> oxides deposited by magnetron sputtering [21], suspensions of Au NPs in methanol or water obtained with the help of laser ablation [18, 19], and Au NPs in glass films formed by the sol-gel method [20], *etc.*

Among the ways of obtaining Au NPs, little attention was paid to pulsed laser deposition (PLD) in vacuum, though one widely and successfully used this method in liquid. It belongs to developed technologies of composite structures and has a number of advantages: congruence of the film composition, flexible control over the parameters of deposition, irradiation of the target, its composition, working-gas pressure, *etc.* [22]. Moreover, the disadvantage of the method consisting in the appearance of drops on the surface of condensates with the formation of rough surfaces can be used for the production of gold films manifesting the absorption at local surface plasmons. As far as we know, one has not yet established the influence of the gold concentration in the target on its PL properties, PL relaxation times, relation between the optical and PL properties of gold nanostructures, *etc.*

The aim of the given work consists in the investigation of the optical and PL properties of gold films and Au NP/Al<sub>2</sub>O<sub>3</sub> (SiO<sub>2</sub>) composite structures obtained by the PLD method in vacuum depending on the conditions of their production.

## 2. Experimental Technique

The gold nanostructures were formed with the help of the PLD method in vacuum using a direct high-energy flux of erosion torch particles, as well as a reverse low-energy flux. In the case of the deposition from the direct particle flux, the films were rough and “nonporous”, from the reverse one – smooth and porous. The investigations performed by the scanning atomic-force microscopy method in the periodic contact mode showed that the grain dimensions on the surface of the films deposited from the direct flux reached 100 nm, whereas the grain size distribution was wide. The scanning electron microscopy technique demonstrated that the grain dimensions of the films deposited from the reverse particle flux

did not exceed 10–20 nm. According to the data of X-ray phase analysis of Au NP/Al<sub>2</sub>O<sub>3</sub> (SiO<sub>2</sub>) composite films, they include an amorphous phase of the corresponding oxide and the cubic and X-ray-amorphous phases of gold. In the case of the deposition from the direct particle flux, glass, quartz, and silicon substrates were placed in normal to the torch axis at a distance of 20–25 mm from the target, while, under the deposition from the reverse flux, they were located in the plane of the target. In the latter case, the films were wedge-shaped in thickness, and NPs were size-selected: larger NPs were deposited close to the torch axis, smaller ones – at some distance. The target represented bits of gold or gold and aluminum (silicon) with a relative share equal to 1–100%. A beam of an YAG:Nd<sup>3+</sup> laser (with a wavelength of 1.06 μm, pulse energy of 0.2 J, pulse duration of 10 ns, and pulse repetition frequency of 25 Hz) scanned the target in a vacuum chamber with the argon pressure amounting to 10–20 Pa. The density of the irradiation energy was varied in the range 5–20 J/cm<sup>2</sup>.

The transmission spectra of the obtained films were measured with the help of an SF-26 spectrophotometer in the wavelength range 340–1000 nm. Some films were thermally treated in air at a temperature of 400–900 °C during 10–60 min. The time-resolved (TR) PL spectra were measured in the energy range 1.4–3.2 eV under the excitation by radiation of a nitrogen laser (with a wavelength of 337 nm and a pulse duration of 8 ns) and the stroboscopic registration of a signal in the photon-counting mode. The strobe width amounted to 250 ns.

## 3. Results and Their Discussion

### 3.1. Transmission spectra of the films

The transmission spectra of the gold films deposited from the direct and reverse fluxes of erosion torch particles included wide characteristic absorption bands related to surface plasmons (Fig. 1, curves 1–4). The minima of the transmission curves belong to the wavelength range 620–700 nm. In the case of the first (rough) films, a decrease of the thickness ( $d$ ) and the irradiation energy ( $j$ ) of the target resulted in a shift of the minima to the blue spectral region (Fig. 1, curves 1–3). The observed regularities agree with the expected ones taking into account that the investigation object represents a rough surface of gold rather than a spherical nanoparticle and that a decrease of both the film thickness and the energy of irradiation of the target reduces the roughness of the films and the size of clusters on their surface. It is known (see, e.g., [1–4]) that, with decreasing the

size of Au clusters, the minimum of the transmission band related to the local plasmon resonance shifts toward the blue spectral region and widens. The widening of the spectra is caused, to a certain extent, by the size dispersion of clusters on the surface of the films. The above-stated facts are also confirmed by the additional study, where the minimum of the transmission spectrum of a gold film annealed for 10 min at a temperature of 400 °C shifted to the short-wavelength region (550 nm) and the absorption band got narrow (Fig. 1, curve 3'). Indeed, it was established that the annealing resulted in the recrystallization of clusters; in the given annealing mode, there take place the deaggregation, decrease of the size of clusters, and smoothening of the film surface.

For the smooth and porous films which were obtained from the reverse particle flux, had a wedge-shaped profile in thickness, and differed in grain size (decreasing with increase in the distance from the torch axis), we observed similar dependences of the transmission spectra on the conditions of deposition (see, e.g., Fig. 1, curve 4). A reduction of the thickness, grain size, and density of the target irradiation energy resulted in a shift of the minima of the transmission bands to the high-energy spectral region. The bands of the films with larger thicknesses, dimensions of clusters, and their dispersion were characterized by a considerable widening. Thus, the gold films obtained under the described conditions of pulsed laser deposition manifested the local surface plasmon resonance. It is explained by the fact that the obtained gold films are characterized by either a well-developed microrelief of the surface or a structure close to that of granulated films due to their considerable porosity.

For the Al<sub>2</sub>O<sub>3</sub> films with Au NPs obtained at an irradiation energy density of 20 J/cm<sup>2</sup>, the plasmon resonance was observed only at the gold concentrations in the target larger than 20% (Fig. 1, curve 5). The minima of the transmission bands belong to the region 550–580 nm, i.e. in a spectral region with shorter wavelengths than that for the gold films. As was expected, as the dimension of Au NPs decreases, the transmission minimum shifts to the short-wavelength side. At the gold concentrations in the target lower than 20%, the dimensions of Au NPs were too small for the manifestation of plasmons. They were also small at irradiation energy densities below 20 J/cm<sup>2</sup>. But, in the latter case, the plasmon resonance bands appeared after the annealing. Moreover, the position of the band minimum did not exceed 540 nm, and the band was symmetric and narrow. This is related to the coalescence, increase in the

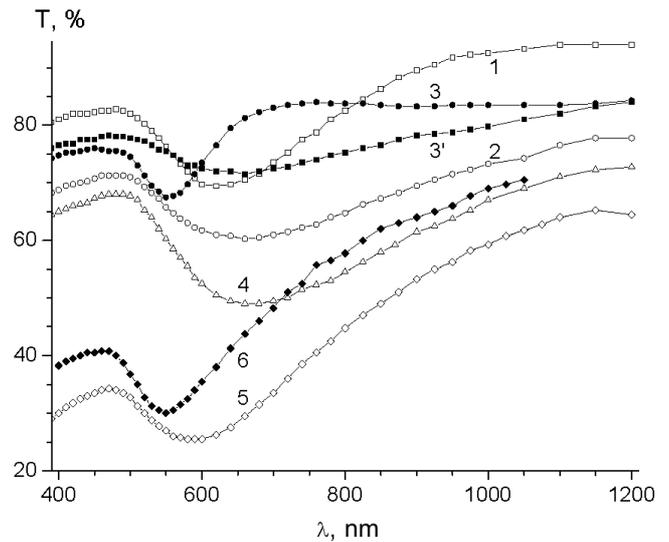


Fig. 1. Transmission spectra of the gold films obtained from the direct (1–3) and reverse (4) particle fluxes, Au/Al<sub>2</sub>O<sub>3</sub> (5), and Au/SiO<sub>2</sub> (6) composite films at deposition conditions differing in thickness  $d$ : 1, 3 – 5 nm, 2 – 10 nm; the energy density of irradiation of the target  $j$ : 1, 2, 4 – 5 J/cm<sup>2</sup>, 5, 6 – 20 J/cm<sup>2</sup>; distance of the substrate from the torch axis  $l$ : 4, 5, 6 – 5 mm; gold concentration in the target  $C_{Au}$ : 5 – 25 %, 6 – 50%. Curve 3' corresponds to the annealed film (3)

dimensions of Au NPs, and approach of their form to a spherical one.

In the transmission spectra of Au NP/SiO<sub>2</sub> films, the plasmon resonance was observed only after their exposure in air during more than two days. This is explained by the fact that the dielectric properties of the matrix change in time not only due to the oxidation of the non-stoichiometric SiO<sub>*x*</sub> phase to SiO<sub>2</sub>, but mainly due to a decrease of the porosity of a film: pores are filled with oxide because of the oxidation of silicon nanocrystals. With increase in the exposure time up to a month, the manifestation of local surface plasmons intensified. The dielectric surrounding of Au NPs had a pronounced effect on the absorption bands. With growth of the time of keeping in air, the minima of the transmission bands shifted to the long-wavelength spectral region. This fact agrees with the known results (see, e.g., [21]) demonstrating that an increase of the dielectric permittivity of a medium surrounding Au NPs results in a shift of the absorption spectrum to the long-wavelength region.

As the share of gold in the target increases from 1 to 10%, the resonance amplitude increases, whereas the minima of the bands shift from 650 to 550 nm and get narrow. In the range of the gold concentrations between 10 and 50%, the positions of the band minima

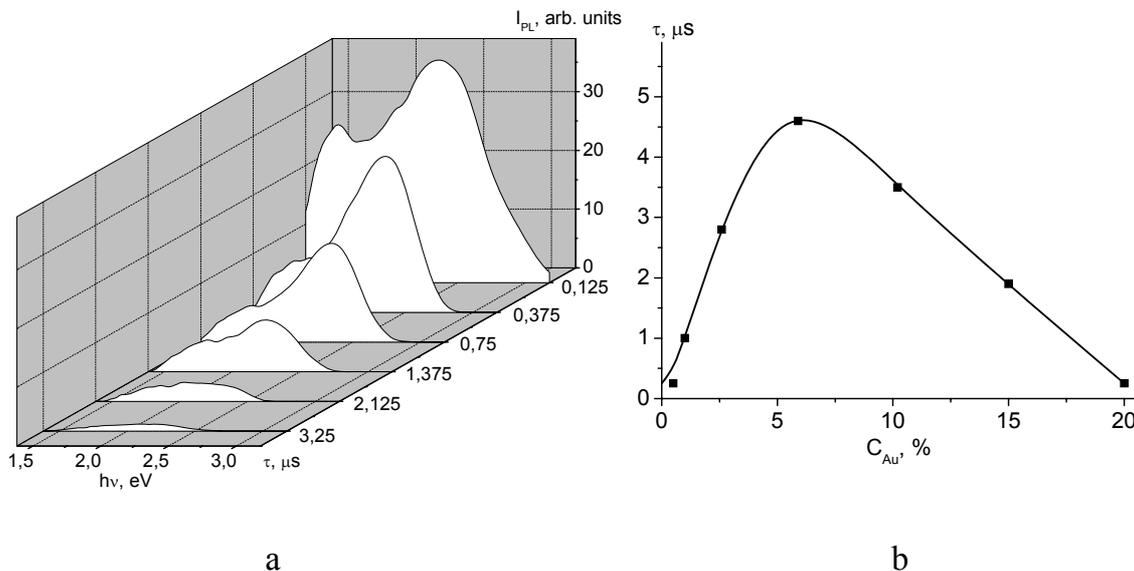


Fig. 2. Time-resolved PL spectra of Au NP/Al<sub>2</sub>O<sub>3</sub> films obtained at  $C_{Au} = 5\%$  (a) and the PL relaxation time as a function of the gold concentration in the target (b).  $P_{Ar} = 13.5$  Pa,  $j = 20$  J/cm<sup>2</sup>

are changed insignificantly. At the gold concentration amounting to several percent, a decrease of the dimensions of Au NPs resulted in a shift of the minima to the blue spectral region.

The influence of the dielectric surrounding of Au NPs on their optical properties is also confirmed by the difference in the behavior of the Au/SiO<sub>2</sub> and Au/Al<sub>2</sub>O<sub>3</sub> films. The Al<sub>2</sub>O<sub>3</sub> matrix is more stable than the SiO<sub>2</sub> one, and the transmission spectra of the films did not change during their storage in air; as was already said, they manifested the plasmon absorption right after the deposition.

### 3.2. Time-resolved photoluminescence spectra of the films

We did not observe PL in the thin gold films obtained both from the direct and reverse fluxes of erosion torch particles. This fact agrees with the information that a very small PL intensity that cannot be usually measured is explained by the radiationless energy relaxation in the case of Coulomb electron-electron scattering that represents a faster process than the radiative recombination of electron-hole pairs accompanying the *sp-d* interband transitions in gold.

In contrast, we observed an intense PL in the case of the introduction of Au NPs into the Al<sub>2</sub>O<sub>3</sub> films. Figure 2, a shows the time-resolved PL spectra for one of the Au NP/Al<sub>2</sub>O<sub>3</sub> films obtained at the gold concentration

in the target  $C_{Au} = 5\%$  and the energy density of irradiation of the target  $j = 20$  J/cm<sup>2</sup>. The analysis of the PL kinetics demonstrates that, if a spectrum contains a high-energy shoulder at 2.9 eV and a low-energy one at 1.6–1.8 eV in the range of the relaxation times  $0 < \tau < 250$  ns, then an increase of the relaxation time results in the abrupt deintensification of the high-energy shoulder, whereas the intensity of the low-energy one decreases slower; at microsecond relaxation times, there remains only a band with a maximum at  $\sim 2.4$  eV and a low-intensity shoulder at  $\sim 1.7$  eV. As far as we know, microsecond PL relaxation times in Au NP / Al<sub>2</sub>O<sub>3</sub> films are observed for the first time.

The effect of deposition conditions on the PL properties of the films obeys the following main rules. First, PL with microsecond relaxation times was observed only in the films that did not demonstrate the plasmon absorption. Second, the dependences of the intensity and PL relaxation times on the gold concentration were correlated and nonmonotonic (see for example Fig. 2, b). At  $C_{Au} < 1\%$ , the times  $\tau$  increased from those lower than 250 ns to 500 ns. With increase of  $C_{Au}$  from 1–2% to 5–6%, the times  $\tau$  grew and reached 5–6  $\mu$ s. In the case of the further increase of the gold concentration up to 15%, the relaxation time  $\tau$  decreased and did not exceed 250 ns at  $15\text{--}20\% < C_{Au} < 75\%$ . Third, PL was also influenced by the irradiation mode of the target. For example, at  $C_{Au}$  amounting to several percent, a decrease of the irradiation energy density from 20 to 5 J/cm<sup>2</sup> re-

sulted in the reduction of the time  $\tau$  from 4–6  $\mu\text{s}$  to less than 250 ns. At  $j = 20 \text{ J/cm}^2$ , PL is intense if  $C_{\text{Au}}$  does not exceed 15–20%, whereas, at  $j = 5 \text{ J/cm}^2$ , it is already weak at  $C_{\text{Au}}$  equal to several percent. PL vanished at  $C_{\text{Au}} = 75\%$  in the case of  $j = 20 \text{ J/cm}^2$  and at  $C_{\text{Au}} = 20\%$  in the case of  $j = 20 \text{ J/cm}^2$ .

The absence of a plasmon resonance in PL films is explained by the fact that free electrons participate in the  $sp-d$  interband transition with the energy  $\sim 2.4 \text{ eV}$  ( $L$  point of the Brillouin zone). It is possible that the low-energy shoulder at 1.6–1.8 eV is related to interband transitions of the  $X$  point of the Brillouin zone, though a contribution of interband transitions to the imaginary part of the permittivity of gold is small for energies lower than 2.06 eV. As for the high-energy low-intensity PL shoulder with small relaxation times, it can be related to nonannealed oxygen vacancies in the  $\text{Al}_2\text{O}_3$  matrix. For the amorphous films deposited from the reverse flux of erosion torch particles without introduction of gold into the target, we observed low-intensity PL with a maximum at 2.9 eV and relaxation times lower than 250 ns. In the case of the annealing in air at temperatures of 800–900 °C during 30–60 min, we succeeded in annealing the aggregates of defects including oxygen vacancies responsible for this high-energy fast PL.

The correlated dependences of the PL intensities and the relaxation times indicate the absence of PL intensification by plasmon local fields. The nonmonotonic dependences of these PL parameters on  $C_{\text{Au}}$  and  $j$  (with regard for the fact that they cause an increase of the dimensions of Au NPs) testify to the existence of the NP dimensions optimal for effective PL. It is possible that, in some Au NPs, there takes place the radiative recombination, whereas, in other ones – the outflow of charge carriers and their scattering on other NPs.

The photoluminescent properties of the Au NP/ $\text{SiO}_2$  films differed from those of the Au NP/ $\text{Al}_2\text{O}_3$  ones. In the case of the deposition of the  $\text{SiO}_2$  films in oxygen at the gold concentration in the target lower than 1–2% in the absence of silicon nanocrystals, the PL spectra were wide and covered the energy range 1.4–3.0 eV, while the relaxation times did not exceed 250 ns (Fig. 3, curve 1). The PL relaxation times of the films deposited in argon slightly changed with increase in the gold concentration in the target to tens of percent (Fig. 3, curves 2–4). The manifestation of oxygen vacancies made a contribution to the high-energy part of the spectrum, whereas that of silicon nanocrystals – into the low-energy one. At  $C_{\text{Au}} = 50\%$ , PL vanished. In contrast to the Au NP/ $\text{Al}_2\text{O}_3$  films, the Au NP/ $\text{SiO}_2$  ones manifested weak PL, and the relaxation times did not reach microseconds.

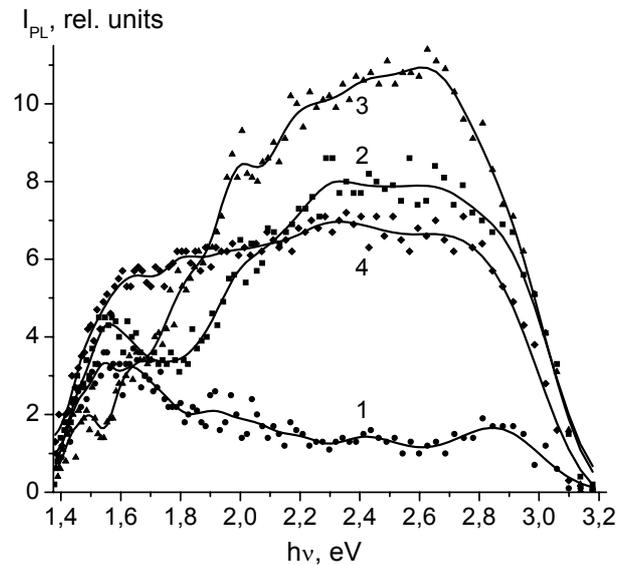


Fig. 3. PL spectra ( $\tau < 250 \text{ ns}$ ) of Au NP/ $\text{SiO}_x$  films obtained at different gold concentrations in the target  $C_{\text{Au}}$ : 1 – 1%, 2 – 5%, 3 – 10%, 4 – 20%; in the oxygen (1) and argon atmospheres (2–4).  $j = 20 \text{ J/cm}^2$

The adduced information testifies to the influence of the dielectric matrix on PL.

The clarification of the PL nature in the considered Au NP/ $\text{Al}_2\text{O}_3$  and Au NP/ $\text{SiO}_2$  composite films requires further investigations. A possible mechanism of the influence of the matrix is related to the dependence of the electron structure of metal NPs not only on the dimensions but also on the oxidation state of metal atoms. The oxidation level of atoms in a simple compound is equal to zero; in gold films, we observe no oxidation, Au(0). Metal complexes include metal ions and ligands – donor particles, anions, or neutral molecules around a metal ion. Along with studying the properties of metal NPs consisting of completely reduced Au(0) atoms, one investigates the properties of metal complexes considering interaction between metal ions [Au(I), Au(II), Au(III)] and organic ligands. It was established [23–25] that the fluorescence is observed in gold clusters consisting of completely reduced atoms, whereas the effective luminescence is characteristic of complexes with gold ions Au(I). It is predicted that, in the latter case, there takes place the hybridization of states of gold ions with organic ligands by means of the formation of charge transfer bands. The luminescence is characterized by radiation with a large Stokes shift and high (microsecond) relaxation times. The optical and luminescent properties of the obtained Au/ $\text{Al}_2\text{O}_3$  composite films at the gold concentration in the target  $C_{\text{Au}} < 20\%$  are similar to

the properties of Au(I) complexes [23,25]. It is possible that the emission represents a result of similar electron transitions, such as interband (*sp-d*) ones together with the charge transfer from the matrix to the metal.

Thus, we have established the conditions and developed the ways of the PLD formation of gold films and composite Al<sub>2</sub>O<sub>3</sub> films with gold nanoparticles, whose optical transmission spectra include extinction bands caused by the local surface plasmon resonance. The manifestation of plasmons in the optical spectrum of the gold films formed by the deposition from the direct and reverse fluxes of erosion torch particles is related to their roughness and porosity, respectively; in the case of the Au NP/Al<sub>2</sub>O<sub>3</sub> (SiO<sub>2</sub>) composite films, it is associated with the dimensions and form of Au NPs, as well as with their surrounding. It is established that PL is absent in the films, where one observes plasmon absorption bands. We determined the conditions for the formation of Au NP/Al<sub>2</sub>O<sub>3</sub> films with effective PL in the range 1.4–3.2 eV with maxima at 2.2–2.4 eV and PL relaxation times reaching 5–6 μs, whose nature is related to interband transitions in Au NPs.

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#### ОПТИЧНІ ТА ФОТОЛЮМІНЕСЦЕНТНІ ВЛАСТИВОСТІ НАНОСТРУКТУР ЗОЛОТА, ОДЕРЖАНИХ ІМПУЛЬСНИМ ЛАЗЕРНИМ ОСАДЖЕННЯМ У ВАКУУМІ

Е.Г. Манойлов

Резюме

Методом імпульсного лазерного осадження в вакуумі сформовано плівки золота та композитні плівки оксидів алюмінію та кремнію з наночастинками золота. Виміряно спектри пропускання та спектри фотолюмінесценції з часовим розділенням у видимій області спектра. Встановлено умови формування наноструктур золота, в спектрах екстинкції яких спостерігається резонансна смуга, що пов'язана з проявом локальних поверхневих плазмонів. На плівках Al<sub>2</sub>O<sub>3</sub> з наночастинками золота, що одержані при концентрації золота в мішені, меншій за 20%, та не мають смуги плазмонного поглинання, вперше спостерігали високу інтенсивність фотолюмінесценції з максимумом смуги при 2,2–2,4 eV та часами релаксації до 5–6 мкс. Обговорено зв'язок між оптичними та фотолюмінесцентними властивостями плівок та природа фотолюмінесценції, що пов'язана з міжзонною рекомбінацією електронів та дірок у наночастинках золота.