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The influence of ultrafast laser processing on morphology and optical properties of Au-GaAs composite structure

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Abstract. The results of direct femtosecond laser structuring of GaAs wafer coated with continuous semitransparent gold (Au) film are presented. The obtained structures demonstrate a combination of different features, namely laser-induced periodic surface structures (LIPSS) on semiconductor and metal film, nanoparticles, Au islands, and fragments of exfoliated Au film. The properties of Au-GaAs samples are studied with scanning electron microscopy (SEM), Raman scattering, and photoluminescence (PL) spectroscopy. The behaviour of phonon modes and enhancement of band-edge PL of Au-GaAs composite sample are discussed. The Raman spectra of Au-GaAs sample processed at different levels of irradiation pulse energy reveal forbidden TO and allowed LO phonon modes for selected geometry of experiment, as well as the manifestation of GaAs surface oxidation and amorphization. A 12-fold increase of PL intensity for Au-GaAs sample with LIPSS compared to initial GaAs surface is observed. The detected PL enhancement is caused by an increase of absorption in GaAs due to the light field enhancement near the Au nanoislands and a decrease of nonradiative surface recombination. The blue shift of PL band is caused by the quantum size effect in GaAs nano-sized features at laser processed surface. The combination of GaAs substrate with surface micro- and nanostructures with Au nanoparticles can be useful for photovoltaic and sensorics applications.

Keywords: laser-induced periodic surface structures, femtosecond laser pulses, gallium arsenide, metal-semiconductor metasurface, Raman scattering, photoluminescence.

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1. Introduction

Gallium arsenide (GaAs) belongs to the actively studied semiconductors, which are effective alternative materials for solar cells and for other applications in photonics and photoelectric devices [1–7]. It has a direct band gap of 1.42 eV and a large refractive index. GaAs demonstrates higher thermal stability and higher electron compared to silicon, which makes it perspective for the absorption and emitting of light. In [1] it is emphasized that GaAs-based solar cells are actively used in space, concentrator solar cells, and military applications due to their flexibility and low weight since they were manufactured as thin-layer solar cells [6]. However, the temperature gradients induce more crystalline defects in GaAs-based devices [1]. Therefore, GaAs began to be used in multi-junction or cascade solar cells in which every layer contains different material that absorbs light in a particular spectral region. Authors of [7] represented GaAs single-junction and multi-junction solar cells (III–V 2-junction, III–V 3-junction solar cells) with potential efficiencies of 30%, 37% and 47%, respectively. The part of defects and impurities, formed during fabrication, are induced by the interface recombination losses, optical losses due to a poor anti-reflection coating, and surface recombination losses caused by the surface states [7].

Surface texturing is one of the standard approaches to increase the effectiveness of solar cells by lowering the surface reflectivity [2, 8, 9]. Moreover, light propagation in the solar element becomes not perpendicular to the plane of the *p-n* junction, consequently increasing charge collection efficiency. A combination of textured semiconductor surface with the plasmonically active metal elements (for example, Au or Ag nanoparticles)

generates an excitation of surface plasmons (SP) in metal nanoparticles or surface plasmon polaritons (SPP) in metal-semiconductor metasurfaces for the efficient light trapping or sensing applications [2, 3, 10, 11].

The application of laser-based processing of GaAs in regimes that induce the highly excited carrier densities and subsequent production of the structures such as laser-induced periodic surface structures (LIPSS) with impressive properties attracts the interest of scientists from both fundamental and application point of view [11–21].

Thus, Margiolakis and co-authors [13] presented a detailed theoretical and experimental study of ultrafast processes and dynamics of the excited carriers under the irradiation of GaAs with femtosecond pulsed lasers. The authors of [15] proposed the fabrication of GaAs micro-optical components of suitable surface quality by wet etching-assisted femtosecond laser ablation. The authors of [16] reported the first terahertz (THz) emitter based on GaAs processed with the femtosecond laser, which demonstrates the enhancement of 65% in THz emission at high optical power as compared to the nonablated material. Both earlier and recent studies [*e.g.*, 17 and 20] have pointed out the critical role of surface plasma electron density in formation of narrow subwavelength nano-ripples. Some scientific groups studied formation of high spatial frequency laser-induced periodic surface structures (HSFL) with a period $<0.3\ \lambda$ in GaAs after irradiation with femtosecond laser pulses in air and vacuum [21–23]. They emphasized the generation and diffusion of point defects induced by the ultrafast laser and showed that the irradiation environment plays an important role in the mechanism of HSFL formation.

In this study, in contrast to the publications in which metal nanoparticles were deposited on the surface of LIPSS after the laser structuring, we perform direct laser processing of a sample consisting of GaAs semiconductor and a thin gold film previously deposited on GaAs wafer.

2. Experimental details

The Au-GaAs metal-semiconductor composite structures have been fabricated on the n-GaAs (100) wafers doped with Te (10^{17} cm^{-3}). The deposition of semitransparent Au films on the surface of GaAs wafers was carried out by the thermal evaporation of a fixed mass of metal in vacuum by using the vacuum thermal evaporation system VUP-5. Before coating the GaAs substrates were degreased, an oxide was removed, and sulfide passivation was performed. The thickness of Au film of about 41 nm was determined using multi-angle incident SE-2000 spectroscopic ellipsometer (Semilab Ltd.).

The ultrafast laser processing of created Au-GaAs composite structures has been performed using a femtosecond laser – a Ti-sapphire laser system based on a Mira-900F femtosecond oscillator with a Legend-HE chirped pulse amplifier (Coherent, USA). The femtosecond laser irradiation had the following characteristics: the central wavelength 830 nm, a pulse duration was within 130…150 fs, a pulse repetition rate was equal to 1 kHz, a pulse energy was close to 0.67 mJ in front of the diaphragm and lens, and 0.27 mJ behind them. A laser power meter "Field Master GS" with a detector head "LM-10" (Coherent, USA) was used to measure the average laser power. The surface texturing has been performed using the scanning beam regime at different distances between the sample and the lens, which leads to different fluences and effective pulse number per spot; and at different velocities in the range 1.3…2.6 mm/s which also leads to a change in effective pulse number per spot. The processing parameters have been close to and slightly beyond the damage threshold associated with the fluences that induce the melting of GaAs [13]. During the laser processing, a vertically standing sample stage moves at a defined velocity. The horizontally polarized laser radiation is incident normally onto the sample surface. The laser processing has been performed in the air in the multi-pulse regime. The irradiation geometry including the focusing by a cylindrical lens and the scanning by the obtained wide beam provides a smooth spatial variation of the irradiation parameters orthogonal to the scan line. In combination with a diaphragm, this allows to create the large areas of homogeneous structures in a single scan, or one can study the effects of different fluences and effective pulse number per spot in different parts of the scan. In this work, the experiment geometry provided the highest fluence and effective pulse number per spot close to one side of the scanned areas (in the upper parts of the scan lines on the Fig. 1) and a decrease in the fluence and effective pulse numbers per spot to the opposite side.

The morphology of obtained Au-GaAs metasurfaces after ultrafast laser processing has been analyzed using a scanning electron microscope (SEM) AURA 100 (SERON Technology Inc., Republic of Korea). 2D Fourier transform (2D-FFT) of SEM images has been used to characterize the obtained periodic structures in the Gwyddion 2.66 software [24].

The Raman spectra of the GaAs wafers and Au-GaAs composite structures have been recorded by RM1000 μ-Raman spectrometer (Renishaw, UK) in the backscattering geometry using the 532 nm Raman laser at room temperature. The main Si phonon peak position (520.5 cm^{-1}) was used as a reference for the Raman measurements.

The photoluminescence (PL) spectra of the Au-GaAs composite sample have been measured using the Acton Research SP-2500i spectrometer with a 600 grooves/mm diffraction grating and a cooled CCD detector under cw 532 nm 50 mW excitation at room temperature. The excitation beam was focused on the sample by using the 100 mm cylindrical lens in an area equal to 0.2×3 mm^2. To eliminate the scattered laser light, a KS-10 long pass filter, which is transparent for wavelengths larger than 600 nm, was placed close to the second lens of the condenser. The samples were attached to a massive metal holder, thus its heating under the excitation beam was negligible.

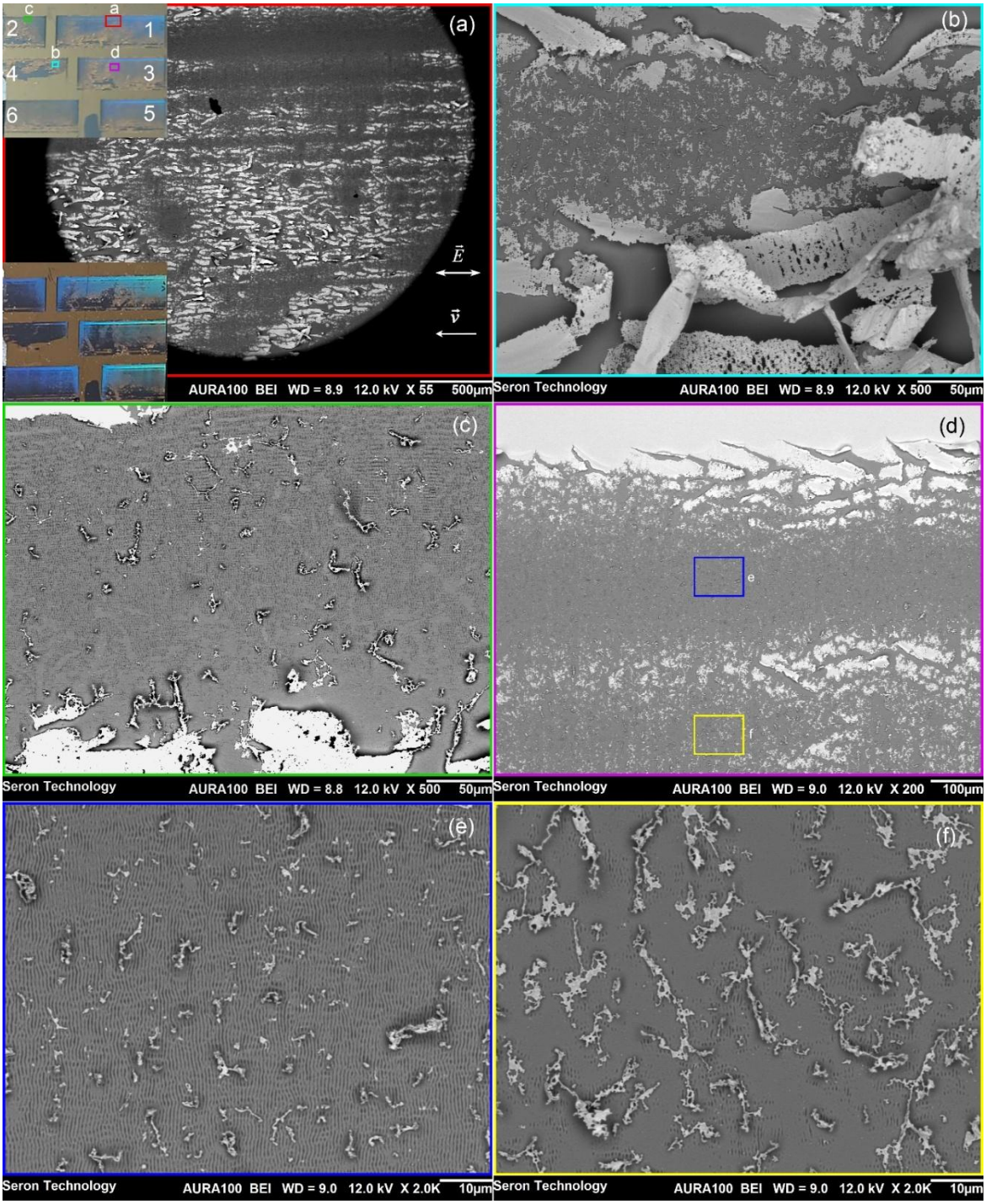

Fig. 1. SEM images of several parts of the Au-GaAs sample with different morphology. The insets of (a) show the photos of the sample under different lighting angles. The structured lines are numerated. Locations of general-view SEM images of the obtained structures (a)–(d) are marked on the sample photo and color-coded. The LIPSS with the Au islands and Au nanoparticles formed in more intensely irradiated part of the line (e) and less intensely (f); their locations are marked in (d).

3. Results and discussion

Fig. 1 presents different characteristic types of the obtained structures. A general view of the obtained structure is presented in Fig. 1a. For all structured areas, the polarization of the laser beam was horizontal and the direction of the laser scanning was from right to left. In the upper part of the scanning lines related to the highest laser fluence, the Au layer is removed, and LIPSS are formed on GaAs with islands of Au and redeposited nanoparticles. This can be observed in Figs. 1a, 1c–1e.

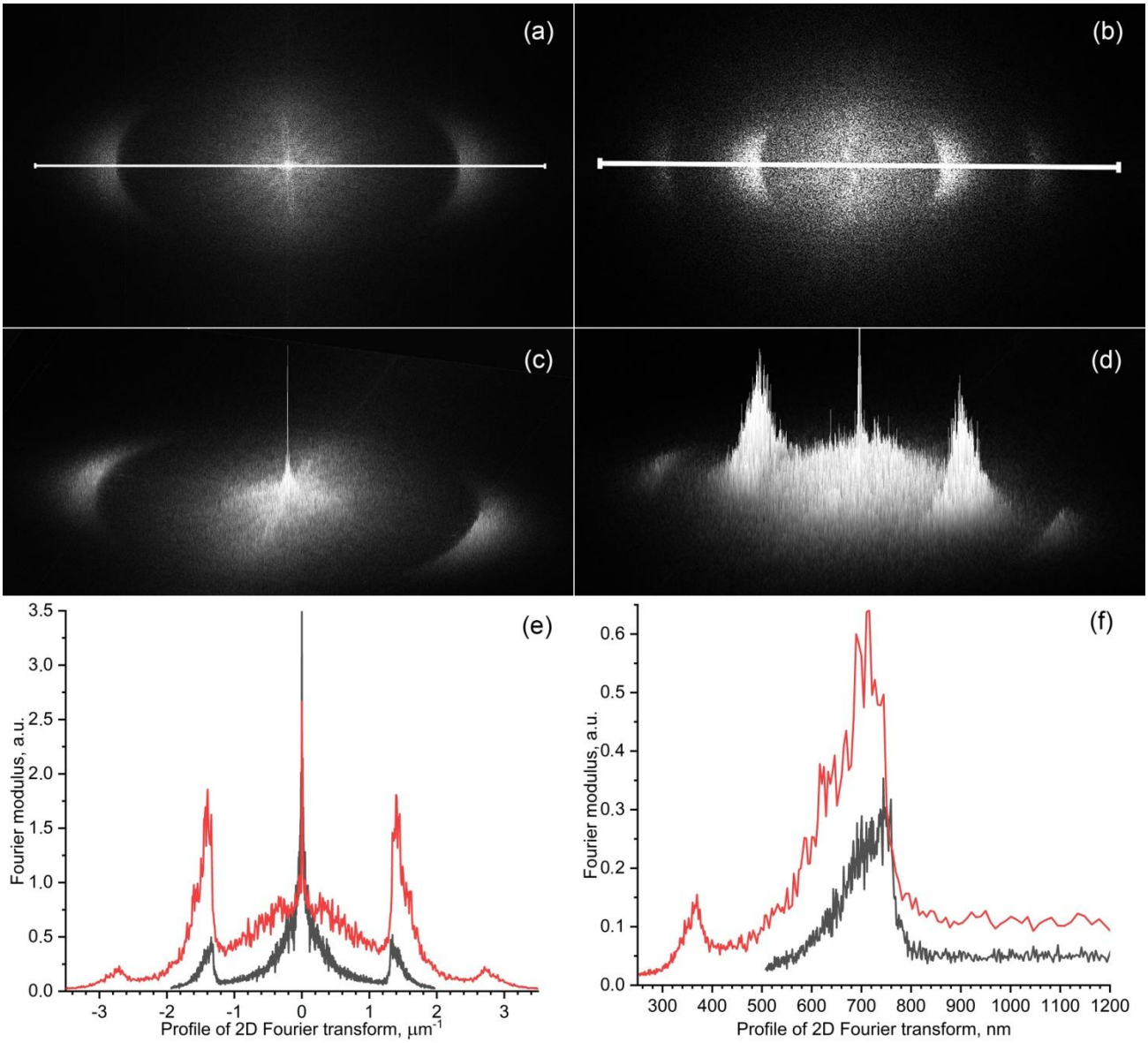

Fig. 2. 2D Fourier transform: of Fig. 1c (a), of Fig. 1e (b). The images are not in the same scale. The corresponding 3D views are presented in (c) and (d). The profiles along the designated directions of the Fourier transforms (a) black and (b) red – (e). Fourier transform profiles in nanometers – (f). For interpretation of the colors in the figures, the reader is referred to the web version of this article.

With decreasing the laser fluence, the LIPSS become fragmented and then disappear, the gold islands become bigger, and the fragments of partially removed and perforated Au film appear (see Figs. 1a–1d, 1f). The strips of perforated and exfoliated Au film are presented in Fig. 1b. The LIPSS and the so-called grooves [13] can be seen in Fig. 1c. The difference in the LIPSS and islands obtained using the slightly different laser fluence can be seen in Figs. 1d–1f. The redistribution of Au nanoparticles and/or Au islands is rather homogeneous for the laser irradiation conditions depicted in Figs. 1c, 1e, and 1f. Fig. 2 shows the results of the 2D Fourier transform of Fig. 1c – Fig. 2a, 2c and black graphs in Figs 2e, 2f and Fig. 1e – Figs 2b, 2d and red graphs in Figs 2e, 2f. The LIPSS period within the range 680…760 nm has been determined. It is worth noting that the reasonable explanation of the LIPSS formation on GaAs is the one that takes into account the conditions of femtosecond laser irradiation that lead to the highly excited carrier densities and further interference of the incident and the surface waves (surface-plasmon (SP) wave) and a fluid-based surface modification mechanism [13]. These phenomena lead to the formation of subwavelength periodic structures oriented perpendicularly to the laser beam polarization.

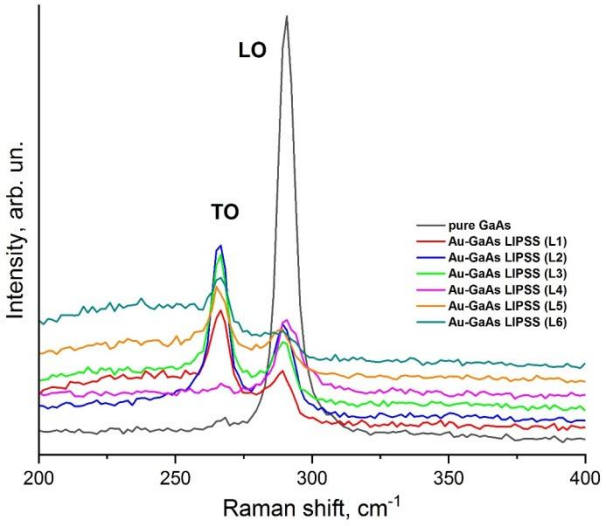

Fig. 3. Room-temperature Raman spectra of initial GaAs (pure GaAs) and laser-structured Au-GaAs composite samples processed with femtosecond laser irradiation at different levels of the pulse irradiation energy density: L1 – 0.115 J/cm^2, L2 – 0.115 J/cm^2, L3 – 0.096 J/cm^2, L4 – 0.072 J/cm^2, L5 – 0.104 J/cm^2, L6 – 0.104 J/cm^2. The excitation wavelength was 532.0 nm. For interpretation of the colors in the figure, the reader is referred to the web version of this article.

Raman spectroscopy has been used as an effective and non-destructive method to study the influence of powerful laser irradiation on the formed Au-GaAs composite structures. The Raman spectra of *n*-GaAs (100) wafers, GaAs LIPSS, and Au-GaAs LIPSS composite sample contain the main bands associated with the transversal optical (TO) phonon mode (~ 266 cm^{-1}) and the longitudinal optical (LO) phonon mode (~ 289 cm^{-1}) of the Γ critical point of the Brillouin zone [25]. The selection rules for Raman scattering in *n*-GaAs semiconductor with (100) orientation and the quasi-backscattering geometry of the experiment predict that only the LO mode should be observable. The Raman spectrum of initial GaAs consists of a strong LO mode and a vanishingly weak TO mode. The TO mode has been revealed in the Raman spectra of all samples of GaAs wafer with Au film processed with femtosecond laser (see Fig. 3).

As we can see, in the Raman spectra of the laser-processed parts of the sample the intensity of TO mode is often larger than LO-mode. The appearance of the forbidden TO-mode indicates the violation of the selection rules for phonon modes for some reasons. In general, the distortion of the back-scattering geometry of the experiment due to the ripples on the GaAs surface and the appearance of defects in the crystal lattice after the laser processing can violate the selection rules.

In the Raman spectra of some parts of the Au-GaAs LIPSS, the wide band within the range 200…245 cm^{-1} has been observed. The nature of this band is determined by the complex influence of surface oxidation, in particular by the formation of oxides (Ga_2O_3 [26–28], As_2O_3 [29, 30], and the amorphous layer of GaAs [31]. The formation of GaAs nanocrystals also induces the low-frequency shift of the phonon peak positions due to the quantum-size effect on phonons in GaAs nanocrystals [32, 33]. At the same time, the main feature of the obtained data is that the crystallinity of GaAs does not undergo essential changes. It should be noted that in the conditions of changes of the electron subsystem of GaAs under powerful ultrafast laser processing, it would be interesting to study the dynamics of electron-phonon coupling, but a detailed presentation of specific peculiarities of Raman scattering is beyond the scope of this paper.

The observed PL spectra contain a single asymmetric band at 870 nm (Fig. 4). Due to the small binding energy of exciton in GaAs, this band observed at room temperature is attributed to the band-edge electron-hole recombination [32]. Its asymmetric shape is caused by energy dependence of the density of states and the charge carrier distribution. The Au-GaAs sample with LIPSS demonstrates a 12 times higher PL intensity as compared to the initial GaAs surface. This large enhancement cannot be explained by the decrease in specular reflection of rough surface of the laser-processed sample. It can be attributed to an increase in the absorption caused by light field enhancement close to gold nanoislands and a decrease in the nonradiative surface recombination. The blue shift of the PL band from 873 nm for GaAs to 870 nm for Au-GaAs sample with the LIPSS and its broadening likely is caused by the quantum size effect in nano-sized features on the laser-processed surface.

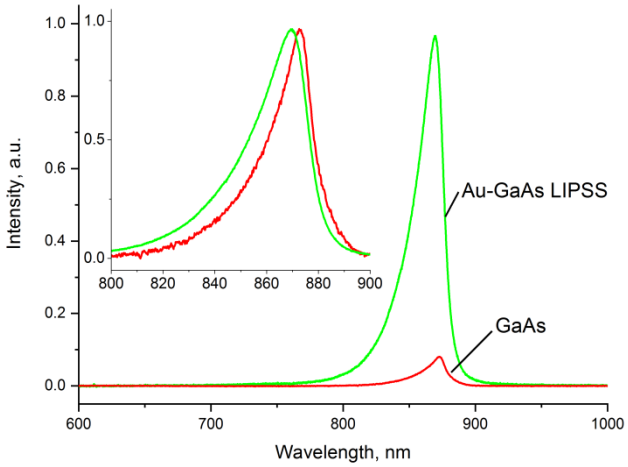

Fig. 4. Room-temperature PL spectra of initial GaAs and Au-GaAs composite sample with the LIPSS. The excitation wavelength was 532 nm. Inset presents the shape of band-edge luminescence peak with intensity multiplied by 12 for GaAs. For interpretation of the colors in the figure, the reader is referred to the web version of this article.

4. Conclusions

The direct ultrafast laser structuring of GaAs wafer coated with continuous semitransparent gold film has been achieved using the linearly polarized femtosecond laser pulses with the wavelength 830 nm. The morphology of resulting structures demonstrates a combination of different features, namely laser-induced periodic surface structures with a period within the range 680…760 nm, depending on the fluence of the laser beam, Au nanoparticles and Au islands, fragments of the exfoliated and punctured Au film.

The Raman spectra of the Au-GaAs composite structures revealed forbidden TO phonon mode besides LO phonon mode allowable for pure *n*-GaAs in the used experimental geometry and a wide complex band within the range 200…245 cm^{-1}. These features can be caused by formation of oxides, an amorphization of GaAs surface, formation of nanoparticles on the surface of the laser-processed sample. The homogeneous distribution of Au nanoparticles and Au islands on GaAs ripples makes the obtained metasurfaces perspective for SERS applications.

The substantial enhancement of more than an order of magnitude of band-edge PL of the Au-GaAs composite sample with LIPSS indicates more efficient charge carrier generation and reduction of their surface recombination that can be useful for photovoltaic applications.

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Dmytruk A.M.: conceptualization, methodology and realization of femtosecond laser processing of Au-GaAs samples.

Вплив надшвидкої лазерної обробки на морфологію та оптичні властивості композитної структури Au-GaAs

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Анотація. Представлено результати прямого фемтосекундного лазерного структурування підкладки GaAs, покритої півпрозорою плівкою золота (Au). Отримані структури демонструють поєднання різних поверхневих особливостей, а саме: лазерно-індуковані періодичні поверхневі структури (ЛІППС) на напівпровіднику і металевій плівці, наночастинки, острівці Au, фрагменти відшарованої плівки Au. Властивості оброблених зразків Au-GaAs вивчали методами скануючої електронної мікроскопії, Раманівського розсіяння та спектроскопії фотолюмінесценції (ФЛ). Обговорюються особливості поведінки фононних мод і посилення крайової ФЛ композитного зразка Au-GaAs. У Раманівських спектрах зразка Au-GaAs, обробленого при різних рівнях енергії імпульсного опромінення, проявляються заборонені ТО і дозволені LO коливальні моди для вибраної геометрії експерименту, а також є прояви окиснення та аморфізації поверхні GaAs. Зафіксовано 12-кратне збільшення інтенсивності ФЛ для зразка Au-GaAs з ЛІППС порівняно з необробленою поверхнею GaAs, що може бути пояснене збільшенням поглинання в GaAs внаслідок підсилення світлового поля поблизу нанострівців Au і зменшенням безвипромінювальної поверхневої рекомбінації. Блакитний зсув смуги ФЛ зумовлено квантово-розмірним ефектом у нанорозмірних елементах на обробленій лазером поверхні. Поєднання підкладки GaAs з поверхневими мікро- і наноструктурами з наночастинками та/або нанострівцями Au може бути корисним для фотовольтаїчних і сенсорних застосувань.

Ключові слова: лазерно-індуковані періодичні поверхневі структури, фемтосекундні лазерні імпульси, арсенід галію, метаповерхня метал-напівпровідник, Раманівське розсіювання, фотолюмінесценція.