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FORMATION OF PERIODIC STRUCTURES ON THE SOLID SURFACE UNDER LASER IRRADIATION

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Advances in the development of technologies aimed at the production of periodic structures on the surface of semiconductors, metals, and insulators have been reviewed. Particular attention was paid to the formation of periodic structures under laser irradiation. The results of both theoretical calculations and experimental researches of the phenomenon concerned are presented.

Keywords: laser-induced periodic structures, laser annealing, temperature profile, nanocrystals.

1. Introduction

Nowadays, laser technologies find a wide application in various technological processes of microelectronics. For example, lasers are used in lithography, for the thin-film deposition, etching, doping, epitaxy, gettering of structural defects, *etc.* [1–9]. In the last decade, lasers were also widely applied as a tool for a structural modification of various semiconductor materials, including silicon as the basic material of microelectronics [10, 11]. In this case, the laser technology was used to produce silicon structures with characteristic micro- and nanoscale inclusions. The obtained structures possess new electrophysical and optical properties, which are different from those in bulk silicon. This circumstance allowed them to be used as a material for fabricating the elements for silicon-based photonics.

Laser technologies for the material treatment are widely used throughout the world in order to create complicated topological two- and three-dimensional micro- and nanostructures in various materials. The interaction of laser radiation with a substance results in the formation of surface structures of various types

such as ripples [12], a wavy relief [13, 14], and micro- and nanostructures [15, 16] in semiconductors, metals, and insulators [17]. The surface structures are formed, as a rule, at the center of a laser spot; they are periodic and arranged with a step varying from hundreds of nanometers to several micrometers [1, 18].

The precision laser drilling [19] and the fabrication of grooved micro-structures [20] comprise a bright example of the application of laser radiation with ultrashort powerful pulses. The method of formation of periodic micro- and nanostructures on the surface at the interference of the incident and surface electromagnetic waves [21, 22] is also widely used. Of particular interest is the capability to form structures, whose dimensions are much smaller than the laser wavelength [23–25]. Surely, the emergence of such structures is possible due to the phenomenon of substance self-organization as a result of nonequilibrium processes running on the irradiated surface. The research of those processes is important from both the fundamental viewpoint (the interaction between the substance and an ultrashort pulse of laser radiation) and the prospects of their application in laser-based technologies [1].

The method of laser interference lithography allows polymeric structures to be obtained with a periodic

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configuration that is the most required and promising now: photonic crystals [26]. A periodic pattern on the polymer surface is of interest for various application domains, including the self-organization in sputtered gold coatings and the interaction with biological cells [27, 28]. The application of the method of modification of a substance under the action of several coherent beams of ultra-violet laser radiation that interfere in the region of their action on the surface of a treated material makes it possible to create structures less than 100 nm in dimensions. The structures of this type are of fundamental and applied interests. In the latter case, we should mark the creation of phase masks in transparent insulators and Bragg fiber-optical and planar reflectors with various spectral characteristics. The capabilities of the method of modification by the interference of laser beams are studied in two directions: (i) the modification of the specimen surface with the use of a photoresist followed by a complete cycle of the lithographic technology (development and etching); (ii) the one-stage modification by the direct action of laser radiation on the surface. In the former case, continuous and pulsed laser sources are used; in the latter one, powerful pulsed lasers are applied [29].

The mechanisms giving rise to the emergence and the development of periodic surface structures (PSSs) under the action of pulsed laser radiation with pulse durations ranging from 10^{-3} to 10^{-11} s, i.e. when this parameter substantially exceeds the characteristic time of electron-phonon relaxation for the irradiated material, have been studied rather well. In those cases, the modulation of a surface relief is formed, when the energy of a laser pulse is absorbed. As a rule, this modulation remains preserved after the pulse action termination. The period and the orientation of a created PSS substantially depend on the laser radiation parameters: the polarization, frequency, incidence angle θ , and pulse energy. Numerous experiments with the application of high-speed diagnostics methods showed that the transformation dynamics of periodic structures in time is characterized by the growth of instabilities. The evolution of the latter has much in common with instabilities taking place at forced scattering and well known in nonlinear optics [30].

According to the experimental interference model, the formation of laser-induced PSSs can be schematically described as follows [30, 31].

1. The process begins with the emergence of a periodically modulated interference electromagnetic field in the space near the material surface. The field emergence origin consists in the interference between the incident laser electromagnetic wave and the wave scattered by the real inhomogeneous surface. In this case, random inhomogeneities of the surface relief can be of both static and dynamic characters. In the latter case, one may say about fluctuation surface waves. The interference of the incident laser electromagnetic wave with definite (resonance) components of diffracted waves is the most effective.

2. In the obtained spatially periodic electromagnetic field, the surface and the near-surface layer are heated non-uniformly in space. The corresponding distribution of the surface temperature evidently correlates with the intensity distribution of the interference light field.

3. If the intensity of incident laser radiation is high enough, the non-uniform surface heating can stimulate the sublimation of the material, its non-uniform melting and evaporation, as well as its removal. As a result, the interference relief becomes "remembered".

Surely, the presented speculations can be considered only in a general sense. For a more rigorous and adequate description of this complicated phenomenon, a problem dealing with an inhomogeneous input of the electromagnetic field energy into the rough surface of an irradiated material has to be considered. Laser irradiation is a very promising way for the treatment of a solid surface, which is associated with the features of energy dissipation, because the latter is mainly realized after the action of a laser pulse has terminated. In this regime, the pure ablation takes place, at which both the thermally induced damage of the material surface and the number of splashes and splinters from the melt on the non-uniformly heated and melted surface are minimum.

The branch of physics aimed at the production of structurally modified materials with prescribed properties is intensively developed today. Those substances find wide applications in laser technologies and microelectronics [10]. Therefore, the development of methods for the fabrication of new structurally modified materials and the research of the properties of those structures are of high scientific and practical interests.

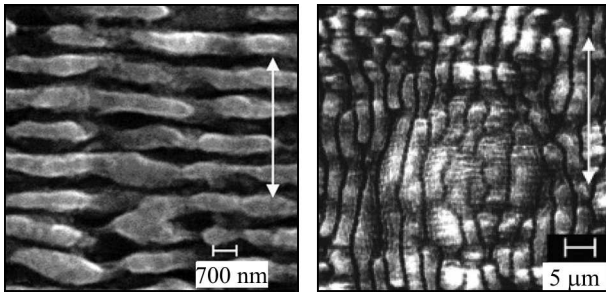
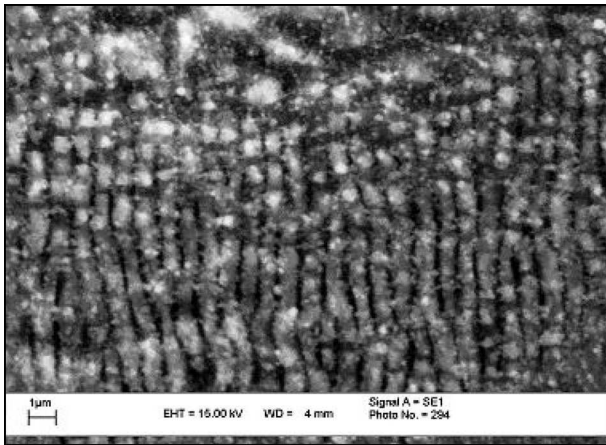
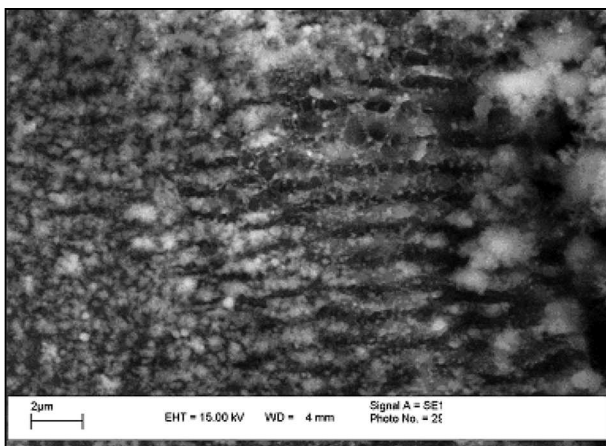


Fig. 1. SEM images of silicon surface irradiated with 1200 laser beam pulses with an energy density of (a) 1 and (b) 2 J/cm². The arrows mark the direction of laser radiation polarization [1]



a



b

Fig. 2. SEM images of Si[100] surface after the laser irradiation with 300 pulses with an energy density of (a) 1 and (b) 2 J/cm² [1]

The aim of this work was to inform the reader about modern developments in the formation of periodic structures on the solid surface with the use of laser radiation.

2. Experimental Researches

The large attention, from both the theoretical and practical viewpoints, is focused on the formation of PSSs in semiconductors. For example, the formation of periodic structures on the surface of the system SiO₂/Si under the action of ultrashort laser pulses was described in works [21, 32]. In those works, polished wafers of single-crystalline silicon were irradiated with pulses generated by a chrome-forsterite laser system with the wavelength $\lambda = 1.25 \mu\text{m}$. The irradiation time amounted to 1–2 min (300–1200 pulses). The pulse duration at the laser system output was 80 fs, the energy density of linearly polarized radiation was varied in an interval of 0.5–2 J/cm², and the pulse repetition frequency was 10 Hz. All experiments were carried out in the air environment and at the normal incidence of a focused laser beam on the specimen surface.

Under the irradiation of polished single-crystalline silicon wafers with femtosecond pulses, the formation of a relief with a period close to the wavelength of laser radiation was observed on the specimen surface. At a laser beam energy density of 0.5–1 J/cm², the obtained PSSs were oriented perpendicularly to the projection of the laser-radiation electric polarization vector. The obtained scanning electron microscopy (SEM) images of the PSSs on irradiated surfaces are depicted in Fig. 1, a. If the intensity of laser irradiation is doubled, the formation of PSSs oriented in parallel to the laser beam polarization vector is observed (Fig. 1, b). However, at a small number of laser pulses, those structures were not distinct, and their formation was unstable (Fig. 2).

The influence of laser radiation on the semiconductor surface can result in the variation of its structural, morphological, and electrophysical properties. These variations depend on the laser radiation parameters, namely, the radiation wavelength and the duration and energy of the laser pulse [10, 33, 34]. A modification in the electrophysical properties of the SiO₂/Si system with the use of the laser irradiation with pulses in the nanosecond interval was observed in the majority of works, when the pulse energy was compa-

rable or higher than the energy, at which the silicon surface melted [35–37].

As one can see from Fig. 3, the orientation of PSSs formed on the semiconductor (Fig. 3, *a*) and metal (Fig. 3, *b*) surfaces under the action of ultrashort laser pulses (USLPs) was always perpendicular to the linear polarization of the laser field. In all works dealing with PSSs that arose on metals and semiconductors under the action of USLPs, the same PSS orientation was observed [1]. The transverse orientation of PSSs was observed only in the cases where the PSS period was shorter or a few times longer than the laser radiation wavelength [30].

The surface ablation in solids subjected to USLPs was analyzed in work [38]. The metal surface was subjected to the action of USLPs, and the following processes took place at that [38, 39]. Laser radiation was absorbed by conduction electrons, and their temperature increased during the action of laser pulses. However, the lattice temperature remained at the initial level. Therefore, the metal, as a system, was deviated by radiation from the thermal equilibrium state and could be regarded as consisting of two subsystems with different temperatures: the electrons at a temperature of about 1 eV and the lattice at room temperature (0.025 eV). The thermal equilibrium between the lattice and the electrons is established within a time interval of an order of picoseconds. If the laser pulse energy was sufficiently high, the surface melted and stayed in this state within nanoseconds. The melt depth amounted to one or a few hundreds of nanometers. After the melt solidification, self-organized PSSs could be detected (see Fig. 4) [38].

The authors of work [38] analyzed various probable mechanisms responsible for the formation of laser-induced periodic surface structures (LIPSSs) under the action of one- or multipulsed femtosecond laser irradiation. The cited authors consider the analysis of the stability of the uniform temperature distributions of electrons and the lattice over the specimen surface plane to be the most promising approach for the consideration of the processes concerned.

In work [40], a method, which can be used for the production of periodic patterns of silicon nanocrystals, was described (Fig. 5). The periodic quasi-one-layer arrays of crystalline Si (c-Si) nanocrystals with relatively identical sizes can be obtained, if the incident laser beam with an appropriate energy density is used for the irradiation. The size of c-Si nanocrystals

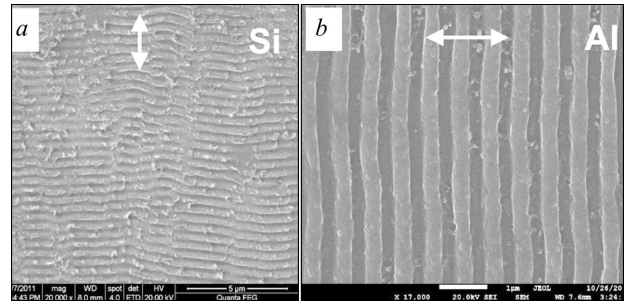


Fig. 3. SEM images of PSSs on (a) silicon and (b) aluminum formed under the action of USLPs with a radiation wavelength of 744 nm [30]

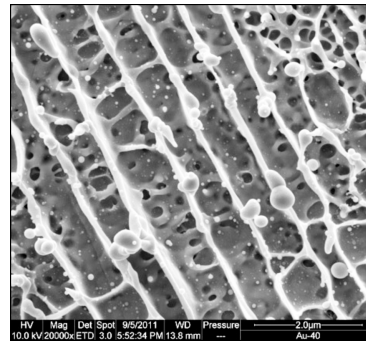


Fig. 4. LIPSS on the gold surfaces formed by a single Ti:sapphire-laser pulse with a wavelength of 800 nm, a pulse duration of 10–13 ns, and an energy density of 3.3 J/cm². The average LIPSS size is about 0.76 μm [38]

was monitored on the basis of the thickness of the amorphous Si (a-Si) layer in the direction perpendicular to the surface. The obtained periodic patterns could be modulated over the lattice [40].

In work [41], the attention was focused on the formation of periodic structures in non-stoichiometric films of SiO_x ($x < 2$). The cited authors analyzed conditions for the production of oxide films using the laser annealing. They demonstrated a possibility to perform the process of patterned (spatially selective) oxidation. Unlike other methods applied to the spatially selective functionalization of the surface – e.g., chemical or plasma treatments, which demand multi-stage procedures (deposition of a masking layer, patterned masks, etching or plasma treatment, mask removal, and so on) – the treatment used by the authors of work [41] is carried out as a one-stage process. Therefore, the spatially selective oxidation of SiO_x films can be done, by using an excimer laser (Fig. 6).

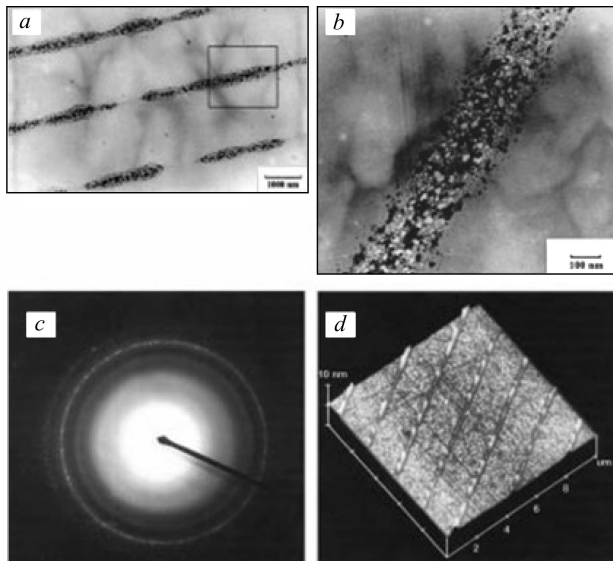


Fig. 5. Transmission electron microscopy (TEM) image of the specimen surface is irradiated through a lattice by an interference laser beam. The energy density in the incident laser beam equals 39 mJ/cm^2 (a). Scaled-up fragment of the surface marked by a rectangle in panel (a) (b). Electron diffraction pattern of the specimen surface (c). Image of the specimen surface morphology obtained using the atomic-force microscopy (AFM) method [40] (d)

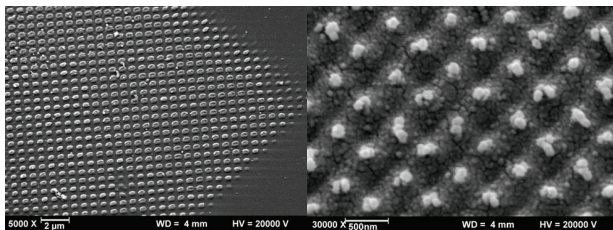


Fig. 6. SEM images of a two-dimensional interference pattern composed of nanoparticles formed structural irradiation of SiO_x film 400 nm in thickness. Parameters of laser irradiation: $\lambda = 193 \text{ nm}$, $p = 100 \text{ mJ/cm}^2$, $f = 5 \text{ Hz}$, and $N = 2000$ pulses [41]

Smooth films with optical quality can be obtained in an atmosphere strongly diluted with oxygen. This kind of treatment is suitable, for example, for the manufacture of optical phase elements, which combine a smooth surface and a varying refractive index. In an oxygen-rich environment, SiO_x films become quickly oxidized. In combination with a high resolution of irradiation, this circumstance can be used to manufacture the arrays of SiO_2 nanoparti-

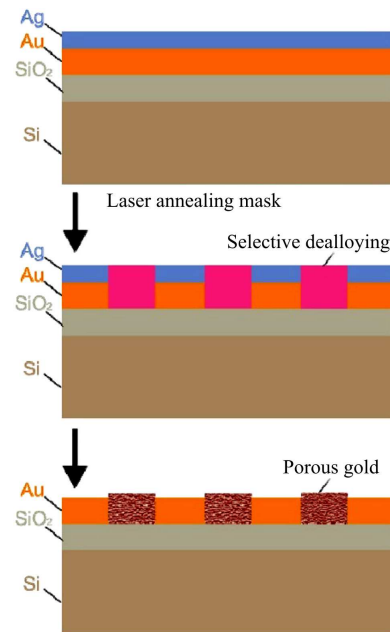


Fig. 7. Schematic illustration of the fabrication procedure of a complex periodic gold structure by combining the laser annealing mask and selective dealloying [42]

cles. In combination with the metallization, these arrays of nanoparticles are promising candidates for the enhancement of surface effects.

Interesting are PSSs on the gold surface. A complex pattern of the Au structure consisting of periodic stripes of nanoporous gold was fabricated, by taking advantage of a combination of the pulsed laser annealing in a mask projection arrangement and the dealloying process [42]. The fabrication of complex periodic gold structures is schematically illustrated in Fig. 7 [42]. Periodic stripes of Ag–Au alloy were produced by the laser annealing of a bilayer (140 nm Ag/80 nm Au) and then transformed into periodic stripes of nano-porous Au with the help of a selective Ag removal from an HNO_3 solution (selective dealloying). Those complex structures had two structural levels: periodic stripes with the period in the micrometer interval and nanoporous gold with a bridge/pore size of about 10 nm.

In Figs. 8 and 9, the SEM images of the morphology of Ag/Au bilayers irradiated with two and ten, respectively, laser pulses with various energy densities in the laser beam are depicted. The width of stripes with a modified morphology increases with the energy density in the beam. At a low density of irra-

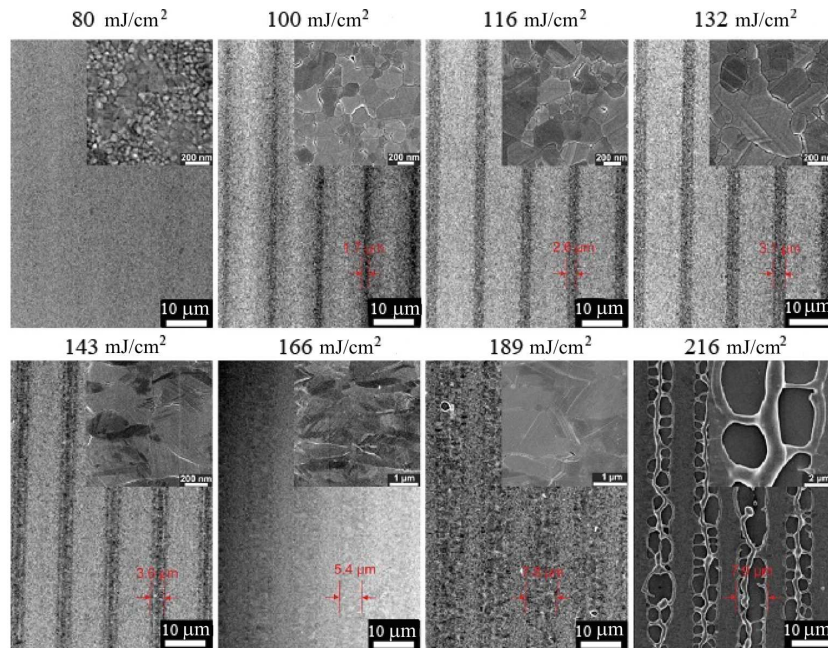


Fig. 8. SEM images of the morphology of Ag/Au bilayers after the laser annealing with the use of two pulses with various irradiation energy densities. The insets demonstrate scaled-up SEM images of the corresponding annealed regions [42]

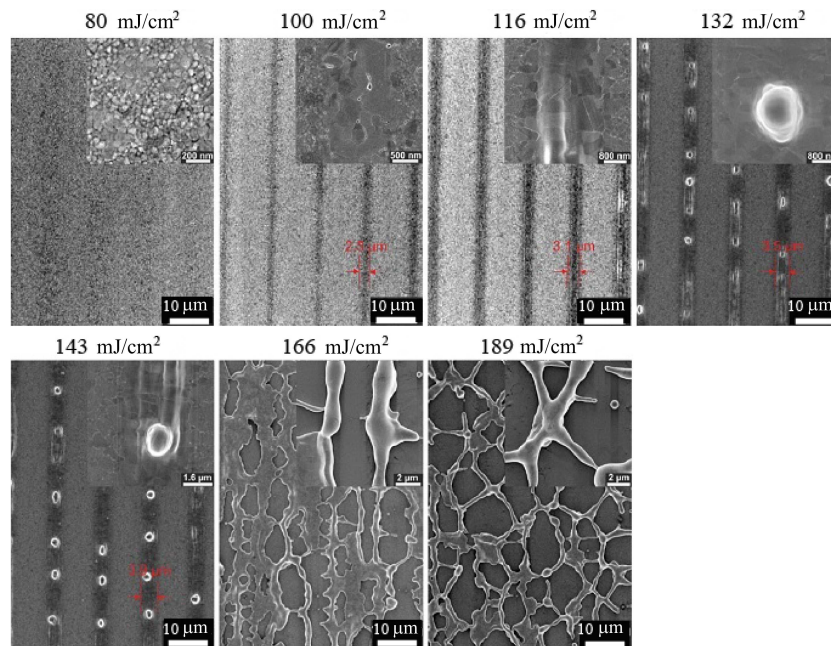


Fig. 9. SEM images of the morphology of Ag/Au bi-layers after the laser annealing with the use of two pulses with various irradiation energy densities (indicated at the top of each image). The insets demonstrate scaled-up SEM images of the corresponding annealed regions [42]

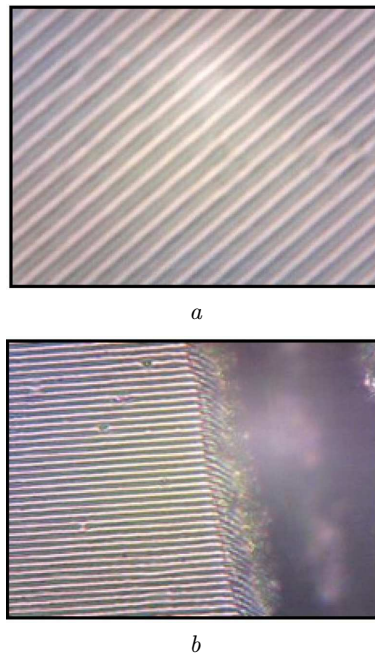


Fig. 10. Micrographs of the surface (a) and cross-section (b) of a polymeric periodic structure with a period of $2\ \mu\text{m}$. The polymer composition is BisA/2Carb 30/70, ZnO 10%; the duration of laser irradiation exposure equals 8 s [26]

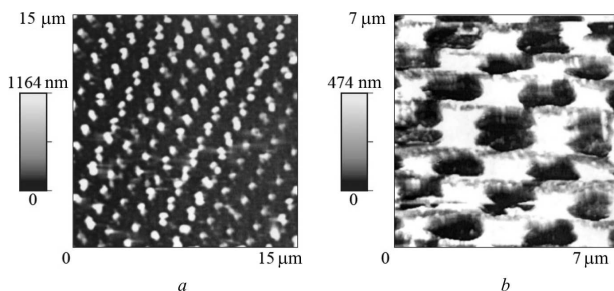


Fig. 11. AFM images of a polyimide film modified by UV radiation of an excimer XeCl laser for various beam energy densities: 60 (a) and 120 mJ/cm^2 (b) [29]

diation energy, heat losses or the heat flux from the irradiation zone result in a narrower modified stripe; whereas at a high energy density, this heat flux gives rise to changes even in non-irradiated regions.

The optimum parameters of the bilayer annealing with 10 pulses is an energy density of laser irradiation equal to 132 or 143 mJ/cm^2 . At the laser annealing, the irradiation energy density required for the effective grain growth must be lower than that for the doping or the formation of Ag–Au alloy in both cases (2 or 10 laser pulses, respectively). For the effective

doping, a higher irradiation energy density or a larger number of pulses is required. A lower energy density is required, when the melting occurs under the action of 10 pulses as compared with 2 pulses (Fig. 9).

Besides the structuring of semiconductors and metals taking advantage of the laser irradiation method, the structuring of polymers deserves a special attention. For example, in work [26], polymeric PSSs were formed, when recording the interference structure formed at the interaction of two plane waves. A helium-cadmium laser with a wavelength of 325 nm was used as a radiation source, which was associated with the absorption band of the applied photopolymerization initiator. UV-compositions on the basis of acrylic monomers and nanocomposites were used as researched materials. It was shown experimentally that periodic polymeric structures can be produced by the method of laser interference lithography in UV-compositions on the basis of acrylic monomers and nanocomposites. In Fig. 10, a, a fragment of the structure surface and a photo of the cross-section obtained with an optical microscope “Labomed-3” at 1000x-magnification are exhibited [26].

The formation of nano-dimensional PSSs in the films of polycrystalline synthetic diamonds and the polyimide films on the fused-quartz substrates under the influence of the nanosecond ultra-violet (UV) radiation from an excimer XeCl laser with a wavelength of 308 nm was studied in work [29]. While studying the laser-induced modifications of polyimide films, it was found that, at an exposure below 100 mJ/cm^2 , there emerged swells on the film surface (Fig. 11). The AFM images of such a fragment is shown in Fig. 11, b. The height of those swells varies from 300 to 1000 nm depending on the exposure dose. At laser irradiation densities more than 100 mJ/cm^2 , there appeared holes in the film. In Fig. 11, b, the AFM image of a modified film surface with holes is shown [29]. An object of this kind can be used as a mask in the submicronic lithography.

For the PSS formation, nanosecond laser pulses are used as a rule. However, recently, researchers started to use picosecond [48] and femtosecond [28, 43–47] laser pulses in their experimental works.

3. Theoretical Researches

Recently, numerical methods are applied more and more frequently, while calculating the PSS period.

Those methods include the creation of surface electromagnetic modes for the given relief geometry. In work [49], by using silicon in the “premetallic” excitation state as an example, the main distinctions were demonstrated between the Sipe model and the numerical method used to solve Maxwell’s equations in the case with a given surface roughness. Namely, it was shown that high-frequency components are determined incorrectly in the Sipe model, and only the averaged rather than actual roughness parameters are considered. Using silicon as an example, those two calculation approaches were compared for various irradiation regimes [50].

In work [51] on the basis of calculations and the comparison of results with experimental data, a conclusion was drawn that the numerical calculation of surface electromagnetic (EM) modes that emerge owing to surface inhomogeneities (cracks, swells, and so on) is required for the one-pulse irradiation regime, whereas the Sipe model can be used at the multipulse irradiation. Again, on the basis of experimental results and a numerical simulation of surface EM modes for various materials, a qualitative explanation was given to a reduction of the PSS period with the growth of the number of laser pulses [52].

A relation between PSS parameters (contrast, groove depth) and main thermodynamic properties of researched materials was studied theoretically and experimentally. In the case of metals, the influence of the hot electron diffusion and the electron-phonon interaction was considered [53]. It turned out that, provided identical irradiation conditions, the stronger the electron-phonon interaction, the deeper are the grooves [30].

In work [54], the numerical calculation of Maxwell’s equations by the finite-element method was performed in the case of the incidence of a plane EM wave on a harmonic surface lattice realized on GaAs with the experimentally measured amplitude $h = 180$ nm, the period $\Lambda \approx 650$ nm, and the dielectric permittivity $\varepsilon = -5 + 20i$. The energy density of laser irradiation was $F = 0.27$ J/cm². The results obtained showed that the absolute value of the squared EM-field strength is approximately 4.5 times larger at the groove surface and only by a factor of 1.5 at the crest in comparison with the plane surface case (Fig. 12). The obtained amplification coefficients multiplied by the incident energy density $F \approx 0.27$ J/cm² give the maximum value for the lo-

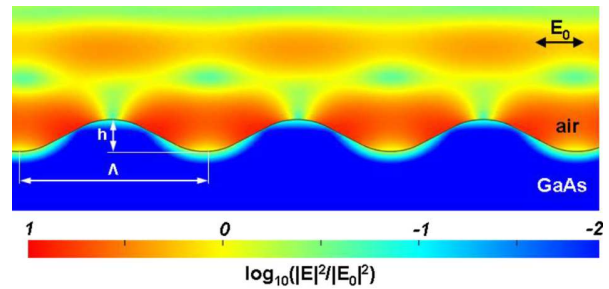


Fig. 12. Calculated distribution of the squared absolute value of the vector of electric field strength \mathbf{E} near the surface of photoexcited GaAs normalized by the incident EM field with the strength E_0 and polarized perpendicularly to the surface lattice orientation [30]

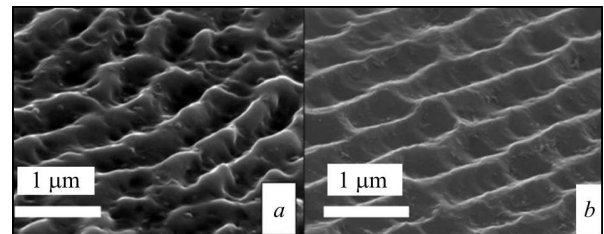


Fig. 13. SEM images of PSSs on the GaAs surface ($F = 0.25$ J/cm²) observed at an angle of 60°: before chemical surface etching (a) and after 30-s and 5-min surface etching with the solution H₂SO₄ : H₂O₂ (3%) : H₂O (b)

cal energy density of EM fields $F \approx 0.4$ J/cm² at the crest and $F \approx 1.2$ J/cm² in the groove.

In comparison with the EM-field energy density threshold for the spallative ablation of GaAs (0.32 J/cm²), the energy density in the groove is much higher. This fact testifies that the dynamics of PSS changes with the variation of the number of pulses has different characters at the crest and in the groove. The presented values of local energy densities may be a little overestimated, because real PSSs on GaAs have plenty of inhomogeneities (Fig. 13), which make a considerable contribution to the energy losses of surface EM waves. Nevertheless, it was important to demonstrate a qualitative pattern of the EM field distribution near the surface [30]. From the aforesaid, a conclusion can be drawn that the heating is more intense in the PSS grooves than at the crests. It also follows that, in the case of multicomponent materials, one may expect the enhanced heating and segregation in those regions.

In work [55], the method was described for the production of clarifying PSSs on silicon with the

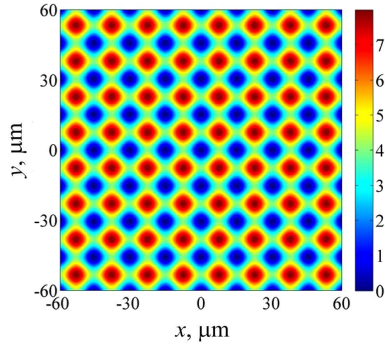


Fig. 14. Simulation result for the intensity distribution in the interference EM field of four laser beams at an angle of 20° and with a period of $15 \mu\text{m}$ [55]

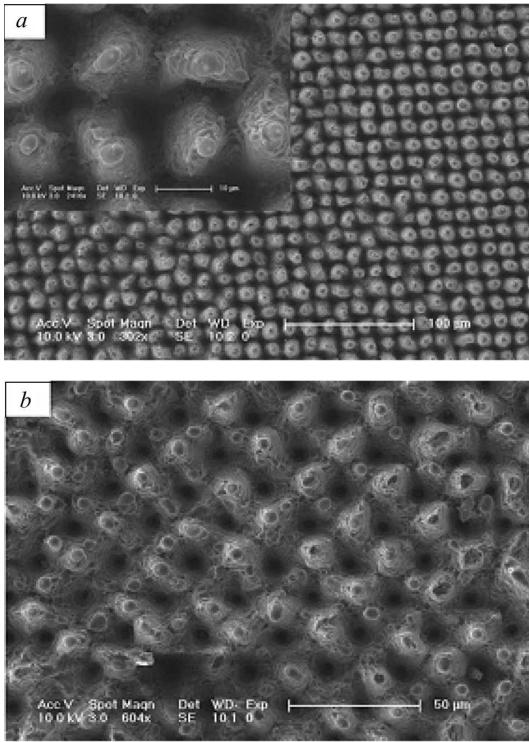
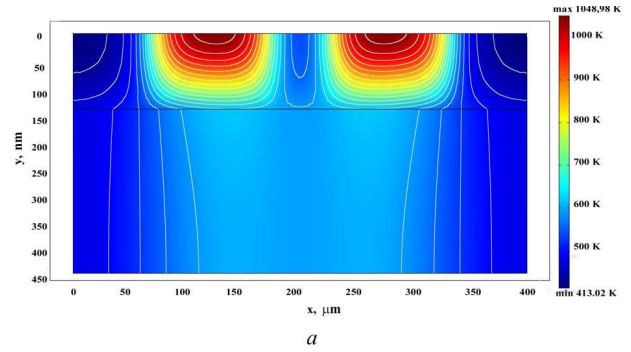
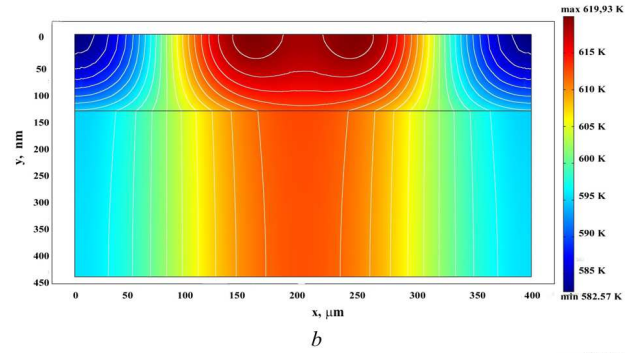


Fig. 15. SEM images of black silicon PSSs which are realized on single-crystalline silicon after the laser irradiation with various numbers of pulses: 600 (a) and 300 pulses (b) [55]

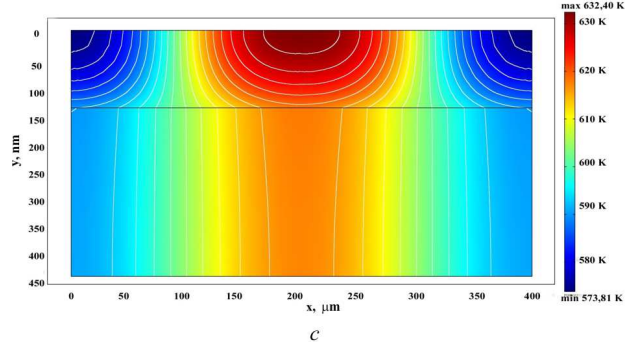
help of the four-beam laser interference lithography (LIL). This method is especially suitable for the fabrication of functional periodic 2D and 3D structures with micro- and nanoscale dimensions. This method made it possible to produce micro-structured black silicon. Being based on the principle of multibeam



a



b



c

Fig. 16. Temperature profiles in the SiO_x specimen at various time moments and at various distances between surface-irradiating laser beams with an energy density of 52 MW/cm^2 and a pulse duration of 10 ns: $d = 50 \mu\text{m}$, $t = 20 \text{ ns}$ (a); $d = 50 \mu\text{m}$, $t = 40 \text{ ns}$ (b); $d = 25 \mu\text{m}$, $t = 40 \text{ ns}$ (c) (d is the distance between the beam centers and t the time interval after the beginning of surface annealing with laser beams [57])

interference, when four coherent laser beams are superimposed, the intensity has a certain distribution in the interference field. The space-time distribution of the electric field strength vector can be written as follows [55, 56]:

$$\mathbf{E} = \sum_{m=1}^N \mathbf{E}_m =$$

$$= \sum_{m=1}^N A_m \mathbf{p}_m \cos(k \mathbf{n}_m \mathbf{r}_m \pm 2\pi\nu t + \varphi_m), \quad (1)$$

where A_m is the amplitude, \mathbf{p}_m the polarization vector, \mathbf{r}_m the vector describing the position, $k = \frac{2\pi}{\lambda}$ the wave number, \mathbf{n}_m a unit vector along the direction of EM-wave front propagation, φ_m the initial phase, and ν the frequency. Figure 14 illustrates a simulation result obtained for the intensity distribution in the interference EM field calculated by formula (1).

The topology of a silicon surface produced following the LIL method was studied, by using the SEM method (see Fig. 15) [55]. Four laser beams, each with a pulse energy of 35 mJ, were directed to the silicon surface at an angle of 20° with respect to it. The laser EM wave was polarized vertically with respect to the incidence plane. The total energy density produced by all four beams in the surface plane amounted to 0.64 J/cm². An array of conical spikes separated by a period of 15 μm was formed on the silicon surface. The diameters of microholes shown in Fig. 15, *b* are much smaller than those exhibited in Fig. 15, *a*. As the number of pulses increases, the hole depth grows, and the spikes become relatively higher. According to the results of theoretical consideration [55], the polarization direction of incident interfering laser beams was found to have a large influence on the intensity distribution. It was also shown that the pulse energy was much lower than in the case of normally focused laser beam owing to the growth of obstacles.

We also tried to calculate the formation of periodic nanostructures on the surface of non-stoichiometric SiO_x films under the laser irradiation. A theoretical research was carried out for the propagation of temperature fields. Their profiles in non-stoichiometric SiO_x films annealed by laser beams with identical intensities were obtained (Fig. 16). The results of calculations of the temperature profile evolution in the near-surface layer of SiO_x specimens performed for various distances between the laser beams irradiating the surface with an energy density of 52 MW/cm² and a pulse duration of 10 ns nanoseconds allow the following conclusions to be drawn. The surface temperature of SiO_x films under the laser annealing with a beam intensity of 52 MW/cm² can reach 1800 K. At the same time, the temperature of the specimen surface in the gap between the laser beams is insufficient to stimulate a phase transition of the SiO_x film

into a nanocomposite SiO₂(Si) film system with silicon nanocrystals. Therefore, according to the temperature profiles exhibited in Fig. 16, silicon nanoparticles will emerge exactly at the positions, where the laser irradiation intensity is maximum [57, 58].

4. Conclusions

The interaction of laser radiation with materials and the formation of periodic structures on their surface is a subject of intense researches. Despite that the issues concerning the mechanisms of PSS formation remain open, LIPSSs have a promising potential for their application in micro-, nano-, and optoelectronics, as well as other domains. In particular, PSSs can be used to manufacture new types of MIS transistors, liquid crystal displays, and solar cells. The pulsed laser irradiation with an energy density below the destruction threshold for the material surface is used to produce spatially periodic structures on the surface and in the near-surface layer of dielectric materials, polymers, metals, and semiconductors.

A large variety of experimental works on LIPSSs stimulated the development and application of various theoretical approaches for their description. The majority of them have a consensus that the type and the periodicity of a surface relief substantially depend on the laser radiation wavelength. This circumstance, first of all, is associated with the interference between the incident and scattered beams, which arises owing to the polarization at interfaces resulting from the surface roughness and the inhomogeneity during the laser irradiation. The LIPSSs induced by the irradiation with femtosecond pulses do not correspond completely to earlier LIPSS models developed for the nanosecond laser irradiation. Therefore, there remain a lot of unanswered questions concerning the mechanisms of PSS formation on the surface of various materials under the action of nano-, pico-, and femtosecond laser irradiation, as well as the methods that are based on them.

1. Huin Cong Tu. Ph.D. thesis *Research and Development of Laser Technology for the Modification of Electrophysical Characteristics of the Silicon-Silicon Dioxide System* (St.-Petersburg National Research University of Information Technologies, Mechanics and Optics, 2014) (in Russian).
2. O.O. Havryliuk, O.Yu. Semchuk, O.V. Steblova, A.A. Evtukh, L.L. Fedorenko, O.L. Bratus, S.O. Zlobin, M. Karl-

- steen. Influence of laser annealing on SiO_x films properties. *Appl. Surf. Sci.* **336**, 217 (2015) [DOI: 10.1016/j.apsusc.2014.11.066].
3. A. Glowacki, S.K. Brahma, H. Suzuki, C. Boit. Systematic characterization of integrated circuit standard components as stimulated by scanning laser beam. *IEEE Trans. Device Mater. Rel.* **7**, 31 (2007) [DOI: 10.1109/TDMR.2007.900056].
 4. F. Beaudoin, K. Sanchez, R. Desplats, P. Perdu, J.M. Nicot, J.P. Roux, M. Otte. Dynamic laser stimulation case studies. *Microelectron. Rel.* **45**, 1538 (2005) [DOI: 10.1016/j.microrel.2005.07.061].
 5. O.O. Havryliuk, O.Yu. Semchuk, O.L. Bratus, A.A. Evtukh, O.V. Steblova, L.L. Fedorenko. Study of thermophysical properties of crystalline silicon and silicon-rich silicon oxide layers. *Appl. Surf. Sci.* **302**, 213 (2014) [DOI: 10.1016/j.apsusc.2013.09.171].
 6. A.V. Dvurechenskii. Pulse oriented crystallization of solids (laser annealing). *Soros Obrazov. Zh.* **85**, 108 (2004) (in Russian).
 7. M. Baumeister, T. Scholz, K. Dickmann, F. Vollersten. Influence of Mie-scattering on high-speed micro-perforation considering brilliant laser radiation. *J. Laser Appl.* **22**, No. 2, 48 (2010).
 8. M.A. Vasiliev, M.M. Nishchenko, P.A. Gurin. Laser modification of the surface of titanium implants. *Usp. Fiz. Met.* **11**, 209 (2010).
 9. R. Varache. Ph.D. thesis *Development, Characterization and Modeling of Interfaces for High Efficiency Silicon Heterojunction Solar Cells* (Technische Universität Berlin, 2012).
 10. A. Medvid, I. Dmitruk, P. Onufrijevs, I. Pundyk. Properties of nanostructure formed on SiO₂/Si interface by laser radiation. *Solid State Phenom.* **131–133**, 559 (2008) [DOI: 10.4028/www.scientific.net/SSP.131-133.559].
 11. A. Medvid, P. Onufrijevs, R. Jarimaviciute-Gudaitiene, E. Dauksta, I. Prosycevas. Formation mechanisms of nano and microcones by laser radiation on surfaces of Si, Ge, and SiGe crystals. *Nanoscale Res. Lett.* **8**, 264 (2013) [DOI: 10.1186/1556-276X-8-264].
 12. A. Borowiec, H.K. Haugen. Subwavelength ripple formation on the surfaces of compound semiconductors irradiated with femtosecond laser pulses. *Appl. Phys. Lett.* **82**, 4462 (2003) [DOI: 10.1063/1.1586457].
 13. B.K. Nayak, M.C. Gupta. Ultrafast laser-induced self-organized conical micro/nano surface structures and their origin. *Opt. Lasers Eng.* **48**, 966 (2010) [DOI: 10.1016/j.optlaseng.2010.05.009].
 14. V. Sava, T.L. Mitran, G. Socol, S. Antohe. Silicon surface structuring by XeCl excimer laser irradiation in atmospheric conditions. *Digest J. Nanomater. Biostruct.* **8**, 61 (2013).
 15. B.K. Nayak, M.C. Gupta, K.W. Kolasinski. Formation of nano-textured conical microstructures in titanium metal surface by femtosecond laser irradiation. *Appl. Phys. A* **90**, 399 (2008) [DOI: 10.1007/s00339-007-4349-2].
 16. L.A. Golovan, I.O. Djun, A.E. Dokukina, S.V. Zaboltnov, A.A. Ezhov, P.K. Kashkarov, N.E. Maslova, I.O. Ostapenko, V.I. Panov, V.U. Timoshenko. AFM investigation of nanoparticles formed on silicon surface by femtosecond laser pulses. *Bull. Russ. Acad. Sci. Phys.* **73**, 39 (2009) [DOI: 10.3103/S1062873809010122].
 17. M. Bolle, S. Lazare. Characterization of submicrometer periodic structures produced on polymer surfaces with low-fluence ultraviolet laser radiation. *J. Appl. Phys.* **73**, 3516 (1993) [DOI: 10.1063/1.352957].
 18. J.F. Young, J.S. Preston, H.M. Driel, J.E. Sipe. Laser-induced periodic surface structure. II. Experiments on Ge, Si, Al, and brass. *Phys. Rev. B* **27**, 1155 (1983) [DOI: 10.1103/PhysRevB.27.1155].
 19. F. Korte, J. Serbin, J. Koch, A. Egbert, C. Fallnich, A. Ostendorf, B.N. Chichkov. Towards nanostructuring with femtosecond laser pulses. *Appl. Phys. A* **77**, 229 (2003) [DOI: 10.1007/s00339-003-2110-z].
 20. T.H.R. Crawford, A. Borowiec, H.K. Haugen. Femtosecond laser micromachining of grooves in silicon with 800 nm pulses. *Appl. Phys. A* **80**, 1717 (2005) [DOI: 10.1007/s00339-004-2941-2].
 21. I.A. Ostapenko, S.V. Zaboltnov, G.D. Shandybina, L.A. Golovan', A.V. Chervyakov, Yu.V. Ryabchikov, V.V. Yakovlev, V.Yu. Timoshenko, V.K. Kashkarov. Micro- and nanostructuring of the crystalline silicon surface under the action of femtosecond laser pulses. *Izv. Ross. Akad. Nauk Ser. Fiz.* **70**, 1315 (2006) (in Russian).
 22. K.E. Lapshin, A.Z. Obidin, V.N. Tokarev, V.Yu. Khomich, V.A. Shmakov, V.A. Yamshchikov. Formation of nanostructures on the silicon nitride surface under irradiation of F[2] laser. *Fiz. Khim. Obrab. Mater.* **1**, 43 (2008) (in Russian).
 23. G. Miyaji, K. Miyazaki. Origin of periodicity in nanostructuring on thin film surfaces ablated with femtosecond laser pulses. *Opt. Express* **16**, 16265 (2008) [DOI: 10.1364/OE.16.016265].
 24. M. Shen, J.E. Carey, C.H. Crouch, M. Kandyla, H.A. Stone, E. Mazur. High-density regular arrays of nanometer-scale rods formed on silicon surfaces via femtosecond laser irradiation in water. *Nano Lett.* **8**, 2087 (2008) [DOI: 10.1021/nl080291q].
 25. C. Radu, S. Simion, M. Zamfirescu, M. Ulmeanu, M. Enculescu, M. Radoiu. Silicon structuring by etching with liquid chlorine and fluorine precursors using femtosecond laser pulses. *J. Appl. Phys.* **110**, 1063 (2011) [DOI: 10.1063/1.3619856].
 26. N.D. Vorzobova, Yu.E. Burunkova, V.G. Bulgakova, I.Yu. Denisjuk, N.M. Kalinin. Preparation of periodic structures in polymeric UV-hardened composite materials by laser interference lithography. *Izv. Vyssh. Ucheb. Zaved. Priborost.* **54**, No. 12, 62 (2011) (in Russian).
 27. J. Heitz, B. Reisinger, V. Fahrner. In *Proceeding of the International Conference on Transparent Optical Networks* (Coventry, 2012), p. 1.

28. E. Rebollar, J. Vázquez de Aldana, I. Martín-Fabiani, M. Hernandez, D.R. Rueda, T.A. Ezquerro, C. Domingo, P. Moreno, M. Castillejo. Assessment of femtosecond laser induced periodic surface structures on polymer films. *Phys. Chem. Chem. Phys.* **15**, 11287 (2013) [DOI: 10.1039/C3CP51523K].
29. Yu.K. Veryovkin, N.G. Bronnikova, V.V. Korolikhin, Yu.Yu. Gushina, V.N. Petryakov, D.O. Filatov, N.M. Biturkin, A.V. Kruglov, V.V. Levichev. Formation of two-dimensional periodic nanostructures on the surface of fused quartz, polyimide, and polycrystalline diamond using the method of pulsed four-beam laser interference modification. *Zh. Tekhn. Fiz.* **73**, N 6, 99 (2003) (in Russian).
30. S.V. Makarov. Ph.D. thesis *Nano- and microstructuring of metal and semiconductor surfaces in the air under the action of femtosecond laser pulses*. (Lebedev Fiz. Inst. Ross. Akad. Nauk, 2014) (in Russian).
31. S.A. Akhmanov, V.I. Emel'yanov, N.I. Koroteev, V.N. Semynogov. Influence of high-power laser radiation on the surface of semiconductors and metals: nonlinear optical effects and nonlinear-optical diagnostics. *Usp. Fiz. Nauk* **147**, 675 (1985) (in Russian).
32. S.V. Zobotnov, I.A. Ostapenko, L.A. Golovan, V.Yu. Timoshenko, P.K. Kashkarov, G.D. Shandybina. Third-harmonic generation from a silicon surface structured by femtosecond laser pulses. *Quant. Electron.* **35**, 943 (2005) [DOI: 10.1070/QE2005v035n10ABEH013011].
33. T. Scheidt, E.G. Rohwer, H.M. von Bergmann, H. Stafast. Charge-carrier dynamics and trap generation in native Si/SiO₂ interfaces probed by optical second-harmonic generation. *Phys. Rev. B* **69**, 165314 (2004) [DOI: 10.1103/PhysRevB.69.165314].
34. P.H. Neethling, T. Scheidt, E.G. Rohwer. Second harmonic generation as a technique to probe buried interfaces: research letter. *South African J. Sci.* **105**, 282 (2009), <http://hdl.handle.net/10520/EJC96944>.
35. V.P. Veiko, A.M. Skvortsov, V.I. Sokolov, Ph. Q. Tung, R.A. Khalecki, E.I. Efimov. Effect of laser irradiation on the structures properties such as SiO₂/Si. *Proc. SPIE* **7996**, 79960S (2010) [DOI: 10.1117/12.889523].
36. Z.Yu. Gotra, S.A. Osered'ko. Control of the surface layer properties in microelectronic technology with the help of laser light. *Zarubezh. Elektron. Tekhn.* **12**, 3 (1985) (in Russian).
37. J. Hlávka, H. Jelínková, K. Hamal, V. Prochocký. Pulsed laser-induced recombination centers in silicon. *J. Appl. Phys.* **56**, 1245 (1984) [DOI: 10.1063/1.334061].
38. E.I. Gurevich, S.V. Gurevich. Laser induced periodic surface structures induced by surface plasmons coupled via roughness. *Appl. Surf. Sci.* **302**, 118 (2014) [DOI: 10.1016/j.apsusc.2013.10.141].
39. S.I. Anisimov, B.S. Luk'yanchuk. Selected problems of laser ablation theory. *Phys. Usp.* **45**, 293 (2002) [DOI: 10.1070/PU2002v045n03ABEH000966].
40. Z. Wang, J. Li, X. Huang. Patterned structures of silicon nanocrystals prepared by laser annealing. *Solid State Commun.* **117**, 383 (2001) [DOI: 10.1016/S0038-1098(00)00463-4].
41. J. Richter, J. Meinertz, J. Ihlemann. Patterned laser annealing of silicon oxide films. *Appl. Phys. A* **104**, 759 (2011) [DOI: 10.1007/s00339-011-6451-8].
42. D. Wang, J. Ihlemann, P. Schaaf. Complex patterned gold structures fabricated via laser annealing and dealloying. *Appl. Surf. Sci.* **302**, 74 (2014) [DOI: 10.1016/j.apsusc.2013.12.066].
43. J. Bonse, J. Krüger, S. Höhm, A. Rosenfeld. Femtosecond laser-induced periodic surface structures. *J. Laser Appl.* **24**, 042006 (2012) [DOI: 10.2351/1.4712658].
44. S. Richter, M. Heinrich, S. Döring. Nanogratings in fused silica: Formation, control, and applications. *J. Laser Appl.* **24**, 042008 (2012) [DOI: 10.2351/1.4718561].
45. I. Mingareev, T. Bonhoff, A. El-Sherif. Femtosecond laser post-processing of metal parts produced by laser additive manufacturing. *J. Laser Appl.* **25**, 052009 (2013) [DOI: 10.1051/mateconf/20130802010].
46. B. Tan, K. Venkatakrishnan. A femtosecond laser-induced periodical surface structure on crystalline silicon. *J. Microech. Microeng.* **16**, 1080 (2006) [DOI: 10.1088/0960-1317/16/5/029].
47. Y. Huang, S. Liu, W. Li, Y. Liu, W. Yang. Two-dimensional periodic structure induced by single-beam femtosecond laser pulses irradiating titanium. *Opt. Express* **17** (23), 20756 (2009) [DOI: 10.1364/OE.17.020756].
48. T. Huynh, A. Petit, N. Semmar. Picosecond laser induced periodic surface structure on copper thin films. *Appl. Surf. Sci.* **302**, 109 (2014) [DOI: 10.1016/j.apsusc.2013.10.172].
49. J.Z.P. Skolski, G.R.B.E. Romer, J.V. Obona, V. Ocelik, A.J. Huis in't Veld, J.Th.M. De Hosson. Laser-induced periodic surface structures: Fingerprints of light localization. *Phys. Rev. B* **85**, 075320 (2012) [DOI: 10.1103/PhysRevB.85.075320].
50. T.J.-Y. Derrien, R. Torres, T. Sarnet, M. Sentis, T.E. Itina. Formation of femtosecond laser induced surface structures on silicon: Insights from numerical modeling and single pulse experiments. *Appl. Surf. Sci.* **258**, 9487 (2012) [DOI: 10.1016/j.apsusc.2011.10.084].
51. G. Obara, N. Maeda, T. Miyanishi, M. Terakawa, N.N. Nedyalkov, M. Obara. Plasmonic and Mie scattering control of far-field interference for regular ripple formation on various material substrates. *Opt. Express* **19**, 19093 (2011) [DOI: 10.1364/OE.19.019093].
52. M. Huang, F. Zhao, Y. Cheng, N. Xu, Z. Xu. Origin of laser-induced near-subwavelength ripples: Interference between surface plasmons and incident laser. *ACS Nano* **3**, 4062 (2009) [DOI: 10.1021/nn900654v].
53. J. Wang, C. Guo. Ultrafast dynamics of femtosecond laser-induced periodic surface pattern formation on metals. *Appl. Phys. Lett.* **87**, 251914 (2005) [DOI: 10.1063/1.2146067].

54. T. Apostolova, A.A. Ionin, S.I. Kudryashov, L.V. Seleznev, D.V. Sinitsyn. Self-limited ionization in bandgap renormalized GaAs at high femtosecond laser intensities. *Opt. Eng.* **51**, 121808 (2012) [DOI: 10.1117/1.OE.51.12.121808].
55. Z. Zhang, Z. Wang, D. Wang, Y. Ding. Periodic antireflection surface structure fabricated on silicon by four-beam laser interference lithography. *J. Laser Appl.* **26**, 012010 (2014) [DOI: 10.2351/1.4849715].
56. M. Ellman, A. Rodríguez, N. Pérez, M. Echeverria, Y. Verevkin, C. Peng, T. Berthou, Z. Wang, S. Olaizola, I. Ayerdi. High-power laser interference lithography process on photoresist: Effect of laser fluence and polarisation. *Appl. Surf. Sci.* **255**, 5537 (2009) [DOI: 10.1016/j.apsusc.2008.07.201].
57. O.O. Havryliuk, O.Yu. Semchuk. Propagation of temperature profiles in non-stoichiometric SiO_x films at two-beam laser annealing. *Fiz. Khim. Tverd. Tila* **15**, 862 (2014) (in Ukrainian).
58. O.O. Havryliuk. Theoretical study on laser annealing of non-stoichiometric SiO_x films. *Khim. Fiz. Tekhnol. Poverkhni* **5**, 461 (2014) [DOI: 10.15407/hftp05.04.461].
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ФОРМУВАННЯ ПЕРІОДИЧНИХ
СТРУКТУР НА ПОВЕРХНІ ТВЕРДОГО ТІЛА
ПІД ДІЄЮ ЛАЗЕРНОГО ВИПРОМІНЮВАННЯ

Резюме

Розглянуто досягнення в області створення технологій отримання періодичних структур на поверхні напівпровідників, металів та діелектриків. Особливу увагу приділено формуванню періодичних структур під дією лазерного випромінювання. Представлено як теоретичні розрахунки формування періодичних структур під дією лазерного випромінювання, так і експериментальні дослідження.