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Development of a metasurface fabrication complex by direct laser transfer with independent displacement of donor-acceptor substrates

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The paper presents the development of a complex for the fabrication of metasurfaces using the laser-induced direct transfer (LIFT) method using a system for the independent movement of donor-acceptor substrates. The analysis of the main parameters of the process is carried out: the energy of the laser radiation, the distribution of the power density of the laser beam, the thickness of the donor film, the parameters of the focusing lenses and the distances between the substrates. The necessity of strict control of each of these parameters is substantiated to ensure stable and reproducible transfer of nanoparticles. The main experimental results demonstrate effective transfer of silicon nanoparticles with an optimal film thickness of about 40 nm, the use of high-aperture lenses to minimize energy parameters, as well as the effect of the donor-acceptor distance on the formation of spherical nanoparticles and the prevention of damage to the acceptor surface. The proposed complex and methodology ensure stable formation of metasurfaces with precise positioning and period control.

Keywords: direct laser transfer, LIFT, laser printing, metasurfaces.

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Introduction

Laser transfer technology is an effective and fairly universal method for obtaining meta-surfaces and 4*D*-printing materials. Today, this technology is being developed at a such a pace that we may expect its structures to be formed at a lithography level. Specifics of this method provides extensive options for fabrication of structures, combination of materials and a variety in the geometry of the formed cells. Laser-induced forward transport (LIFT) is a non-contact method of transporting substances for applications in various fields of physics, chemistry, biology and medicine. Unlike photolithography, the laser-induced approach tends to significantly reduce the time and the number of steps to fabricate the required template [1]. The study [2,3] describes modern technologies for printing functional materials and bioprinting with an analysis of the problems and prospects for the method development, including transfer mechanisms, types of lasers and the materials used. Nanosecond and femtosecond pulses create different mechanisms for the transfer of nanomaterials, and the use of femtosecond pulses provides a more accurate and direct transfer [4]. This method is also successfully used for printing hydrogels with living cells, while keeping high viability and preserving the tissue structure [5,6]. Due to the laser radiation parameters, especially the duration and shape of pulses, it is possible to control the transfer process itself and improve the quality and accuracy of nanoparticles deposition [7–10]. LIFT technology allows to transfer metal films, create high-resolution micropatterns [11], elements of sensitive energy saving microsensors [2,3], conductive inks, which is critical for flexible and wearable electronics [12], 3D microstructures with controlled topology and high functionality [13], polymer composites with various properties and structures [14], graphene patterns that enable creation of functional electronic components [15,16].

Despite the huge interest and involvement of many scientific communities in this sector, there's a number of problems in implementing this operation: the frequency of structures, the use of various materials, as well as efficiency of the transfer operation.

The purpose of this study is to highlight the problem of performing a laser transfer of various materials by independently moving the donor and acceptor substrate. The concept of this method is based on the experience of building similar systems, described in [17–22]. These sources were used as a starting point because of the similar characteristics of the available equipment in relation to the solutions and goals outlined in these studies.

1. Materials and design of experiments

The laser printing process requires the use of high-precision and stable linear transfer systems. The period of the structures depends on parameters of the optics used, donor-acceptor distance, as well as the type of transferred materials. The nature of the structures formed during the laser transfer process, as well as the accuracy of the period reproducibility are influenced by the donor-acceptor transfer system. The laser-induced forward transfer when placing the donor over the surface of the acceptor in a static design limits the period of the transferred nanoparticles. The period is limited by the laser beam exposure area on the donor film surface. During the transfer operation, a crater (about 1 μm) is formed on the donor film, which is significantly larger than the transferred object. A crater is an area with a discontinued surface and no film, which, in case if the beam hits this surface again, will disrupt the stable transfer regime; therefore, the period of the formed meta-surface cannot be less than 1 μm (neglecting the probably of compromised adhesion of the donor film in the laser exposure area), which goes beyond the requirements for the periods of structures operating in the visible optical range. Deployment of an independent donor-acceptor transfer technology opens up the possibility, in addition to reducing the period of the transferred structures close to the size of the transferred structures, of forming meta-surfaces from various materials with a period close to the size of the transferred particles; however, in this case stringent requirements are imposed on the accuracy and reproducibility of the transfer systems.

A general view of the experimental setup developed for the laser-induced forward transfer of nanoparticles from the donor surface to the acceptor surface is shown in Fig. 1.

The described system is made on the basis of a process setup used in [23–25]. A femtosecond (fs) laser setup TETA-10 with a wavelength of 515 nm, a pulse duration of 300 fs, and a pulse repetition rate of 10 Hz was used for irradiation. The test donor-films were used for the fabrication of meta-surfaces: Si (25, 40, 70, 100, 130 nm) and Au (40 nm), deposited on-* the quartz glass (120−300 μm). Acceptor surface — Au (8 nm) deposited on the quartz surface (120 μm). The most successful results of the laser-induced forward transfer were achieved with films with a thickness of 40 nm. The 25 nm films showed lower transfer stability due to the high sensitivity of the position relative to the focal plane of the micro lens used and radiation energy fluctuations. The 70, 100 and 130 nm thick films do not allow a single particle to be formed on the acceptor surface due to the large volume of the ejected material. The donor-acceptor distance was about 10 μm. The samples were placed on a developed vacuum holder, which made it possible to fix various samples without developing specialized equipment. The main requirement for the samples is — the thickness is less than the operating length of the lens used (registration of structures or observation in the dark field). This method provides the installation and disassembly of samples without mechanical impact on both, the precision elements of the setup and the installed samples themselves (donor and acceptor). This effect is especially pronounced when using thin (120−150 μm) quartz substrates. To ensure a reproducible result, a displacement precision of at least 0.1 μm on all axes is required. The minimum displacement in linear planes of the setup was 1 nm with bidirectional repeatability of ±75 nm. The transfer process is monitored in real time using the dark field microscopy method, which is effective for qualitative assessment of the transfer results depending on the color of the scattering points and the size of the resulting particles.

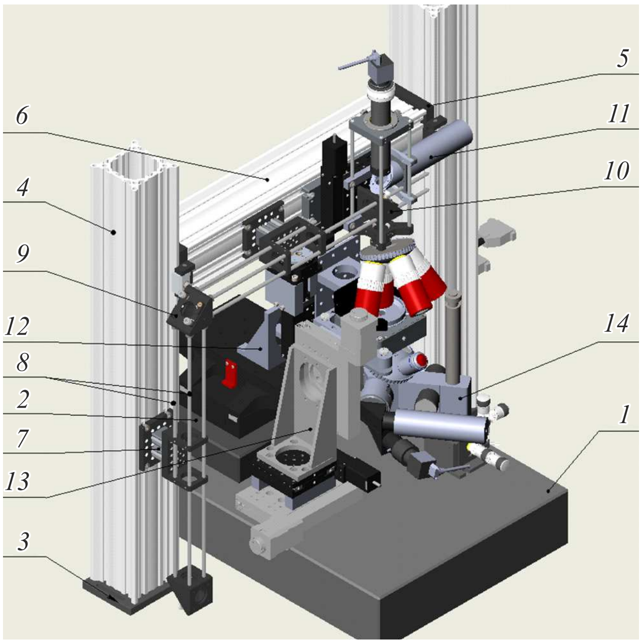

Figure 1. Laser-induced experimental setup with an independent donor–acceptor transfer: *1* — general baseplate of the system, made on a granite calibration plate 400 × 600 × 100 cl.00; *2* — compensation granite calibration plate 300 × 200 × 50 cl.00; *3* — portal mounting support; *4* — vertical support of the portal-periscope radiation transfer system; *5* — bracket of the horizontal guide of the portal-periscope system; *6* — horizontal guide of the portal-periscope system; *7* — mounting spokes attachment to the portal-periscope system; *8* — 6 mm diameter mounting spokes with a length of 450 mm; *9* — kinematic mirror holder with two-wavelength mirror mounted (515 and 1030 nm); *10* — the mounting point of a dichroic mirror is transparent to the visible range, blind to 1030 or 515nm, depending on the radiation used; *11* — laser beam exposure visualization system; *12* — donor movement system made of precision linear guides with a travel of 100 × 100 × 25 mm and a piezoceramic ultra-precision positioner with a travel of 100 × 100 × 20 nm; *13* — acceptor displacement system made of precision linear guides with a travel of 100 × 100 × 25 mm; *14* — transfer visualization system in the dark/bright field fitted with a 50X lens and spectral-selective backlight system.

Within a single technological process several materials may be transferred using the independent donor-acceptor substrates and their separate displacement. Thus, the problem of structures formation is solved regardless of the damage zone on the donor substrate, which, in turn, depends on a set of interrelated factors. This option is deployed in the newly developed complex. General view of the transfer of several materials is shown in Fig. 2.

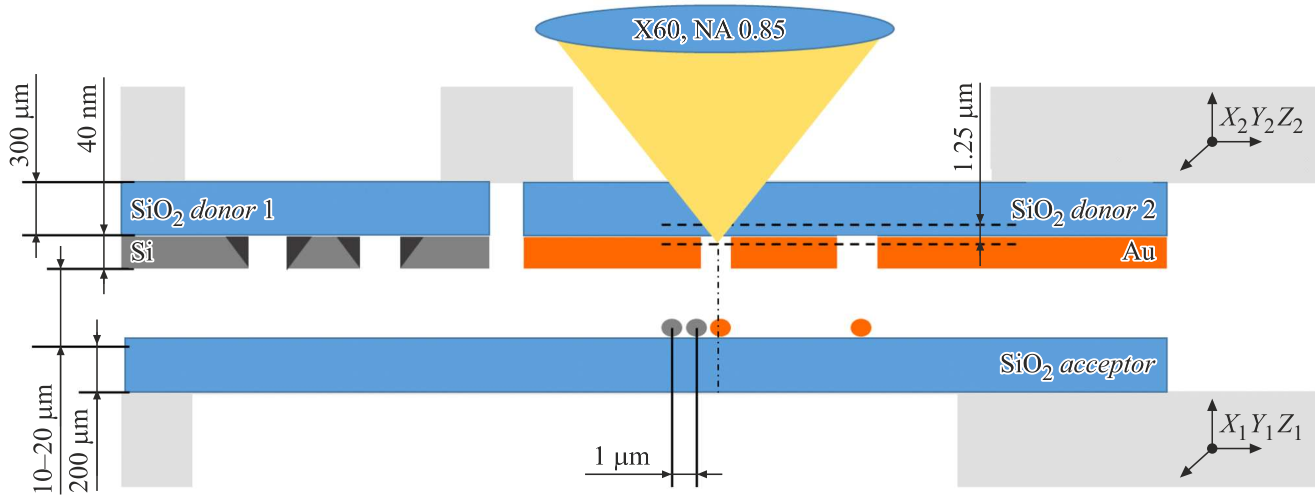

Figure 2. Transfer of several materials within the same technological process.

2. Problems of laser transfer of nanoparticles

Disruption of the structure of the donor film leads to uncontrolled destruction and, as a result, the formation of disordered structures of various sizes and random numbers, which is shown in Fig. 3. The observed defects — is a deviation of the transferred particle direction. This effect can be observed when entering areas of the film that have a stressed state (a zone close to the area of exposure to the laser beam, but visually indistinguishable), as well as when using low-quality films (unstable adhesion of the film to the substrate, thickness fluctuations). The resolution and reproducibility of laser transfer may be improved by removing the steps of treating the film damaged areas by shifting the exposure zone by a distance of units to several tens of micrometers. It is possible to preserve the period of the obtained structures on the acceptor by independently moving the donor substrate relative to the acceptor substrate. This method allows, in addition

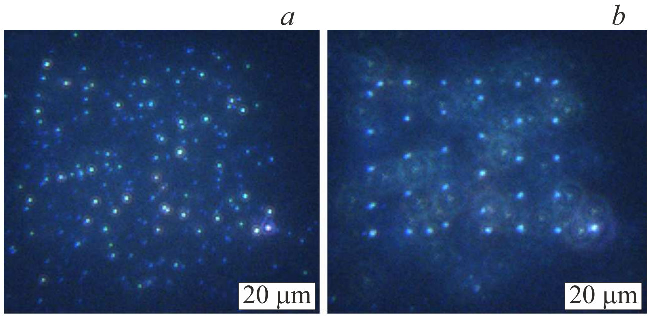

Figure 3. Image by method of dark field microscopy during setting the process of Si transfer onto the surface of acceptor SiO_2: *a* — in the incident/reflected light, *b* — in the dark field.

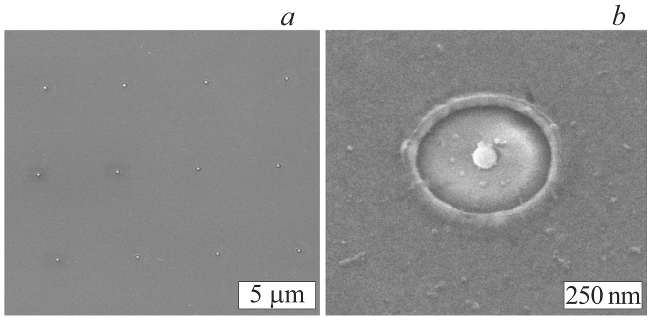

Figure 4. SEM images of laser transfer of silicon nanoparticles to the acceptor surface with a gold film applied: *a* — array of transferred nanoparticles, *b* — image of a single nanoparticle.

to stable transfer, to fabricate the surface using various materials.

The subsequent analysis of the transferred structures, geometry of the particles, formed array, and the number of transferred particles was carried out using the electron microscopy method (Fig. 4). This image shows the result of stable transfer of silicon nanoparticles to the acceptor surface containing a gold film. Diameter of the transferred nanoparticles on the acceptor surface varies within 80–90 nm. These particles were transferred using LOMO 60X NA 0.85 lens with the laser radiation energy of 1–10 nJ in a staggered way with a step of 5 μm, shift of the even row — 1 μm.

The donor-acceptor distance affects the flight time of the particle, which allows the transferred material to transform into a spherical body. During the flight, contact with atmospheric gases allows the ejected material to cool down in the shape of a sphere. Deformation effect oftens occurs during the transfer of „hot“ particles. The shape of the sphere is distorted, as shown in Fig. 5.

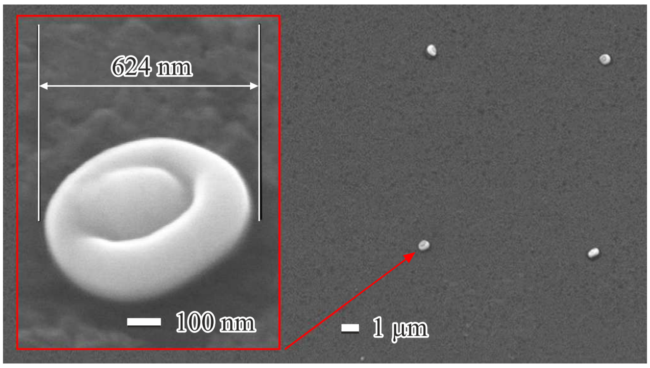

Figure 5. SEM image and insert window showing deformation of the transferred object.

3. Results and discussion

In sec. 3 the prevailing parameters that make the greatest contribution to the process and require special attention during the laser-induced forward transfer are highlighted.

I. The volume of the material to be removed and its condition during ejection depends on the energy of laser radiation. In the process of interaction of laser radiation with the glass-material interface of the donor, successive physical processes may develop depending on the energy increase: acoustic film separation, local melting, plasma formation. The absorption and dissipation of energy of femtosecond laser pulses are nonlinear and depend on the properties and condition of the processed material. In laser processing of thin films, the dimensional effect plays a key role, since the thickness of the film affects both the processes occurring during exposure and the relaxation processes after it. The energy transfer of laser pulses causes an intense displacement of the electron gas inside the material crystal lattice. For the thin films, the energy of the film-subsrate adhesion compared to the energy of vibrations of the lattice atoms (where atomic position shifts reach several lattice periods) leads to the separation of the film from the substrate. In the future, when the separated film moves due to thermal conductivity, the material passes into a molten state, which balances the energy state of the entire ejected cluster. Under the influence of surface tension, the molten cluster assumes a spherical shape. Upon ejection, a number of such spheres may form, depending on the film thickness, initial energy of laser radiation, and power distribution in the laser beam. The ideal result is the formation of a single main sphere with a diameter of several tens to several hundred nanometers. Stable transfer is possible with precise adjustment of the laser radiation energy and maintaining high stability of this parameter.

II. The distribution of power density in the cross-section of a laser beam is one of the key parameters determining the efficiency and accuracy of the laser-induced forward transfer process. The spot should have a strict axisymmetric distribution of the laser energy. Otherwise, it is impossible to achieve the transfer of nanoparticles coaxially with the optical axis of the system. As a result, defects are possible in the form of deflected transferred particles from the optical axis of the system, the formation of an uncontrolled number of transferred droplets on the acceptor surface instead of one droplet. Thus, the quality of the power density distribution in the caustic zone shall strictly correspond to the diffraction quality of the laser beam and be controlled with high accuracy to ensure a coaxial homogeneous transfer of the material.

III. The thickness of the film has a significant effect on the volume of material ejected during laser-induced forward transfer. In this technology, there is a fundamental limitation related to the laser wavelength, which determines the minimum size of the impact area on the donor film while maintaining the required diffraction quality of the beam. In theory, the resolution may reach the values of $\lambda/2$. In this study, laser radiation with a wavelength of $\lambda = 515$ nm was used, forming an impact spot with a diameter of about 250 nm, which corresponded to the potential limit of spatial resolution of the transferred material. Thus, it is possible to adjust the ejected volume by changing the film thickness.

IV. The focusing lens shall ensure formation of a laser spot of minimal size and diffraction quality at the substrate-donor film interface. The lens used shall transfer sufficient energy to initiate the separation of a minimum portion of the material, while preventing excessive volumetric heating. The use of lenses with a high numerical aperture makes it possible to significantly reduce the length of the focusing zone (caustic zone), reducing the laser beam waist. This helps to concentrate energy in the transferred material, minimizing inhomogeneities and separation of the ablated film area into multiple objects.

The beam waist parameters when focusing with different lenses were estimated using the knife method at 10%/90% level of power, the knife shift step was 50 nm. The measured values deviate from the theoretical values ($D_{Airy} = 1.22 \cdot \lambda/\mathrm{NA}$) by no more than 10%. The waist length (Rayleigh length) was determined analytically according to the ratio ($Z_R = \lambda/\pi \cdot \mathrm{NA}^2$). The values of Rayleigh length and waist length are shown in the table. It should be underscored that in LIFT in the described configuration of the experimental setup, controlled transfer was carried out using a LOMO PLAN-APO microobject 60x/0.85. Probably, the result is due to the small waist length formed by this micro lens compared to lenses with a smaller numerical aperture. The use of micro lenses with a numerical aperture of up to 0.85 did not allow the formation of single particles on the surface of the acceptor substrate.

V. The distance between the substrates in the process of laser-induced forward transfer, according to the literature data, should be in the range of $10-200\,\mu$m. An increase in this distance leads to a significant deviation of the trajectory of the transferred nanoparticles from the laser radiation axis, which may exceed the permissible offset relative to a period of the formed structure. At distances less than $10\,\mu$m, there is a high probability of damage to the acceptor substrate

Lens parameters and laser beam areas

Micro-lens	Waist diameter, μm	Waist length, μm	Operating section, mm	Transmittance, % (at 515 nm)
Mitutoyo M Plan Apo Nir 50x/0.42	1.1	1.86	17	71
Mitutoyo M Plan Apo Nir 100x/0.50	0.8	1.33	12	42.5
Mitutoyo M Plan Apo Nir HR 100x/0.70	0.75	0.7	10	48
LOMO PLAN-APO 60x/0.85	0.75	0.46	0.45	55

surface by laser radiation, which is especially critical for acceptors with thin film coatings, as illustrated in Fig. 4, *b*.

VI. The donor film exposed to laser radiation shall be isotropic to ensure uniform adhesion to the substrate in all areas of the film, as well as maintain a constant thickness over the entire surface. Each of these parameters has a critical impact on the stability and reproducibility of laser transfer processes. Deviations from the set parameters result in a less stable transfer regime or make it impossible to determine adequate parameters for the predicted transfer of material from the donor to the acceptor.

Conclusion

The setup developed as part of the study allowed a stable laser transfer of single particles from a donor substrate (silicon) to an acceptor surface (a silicon oxide substrate with a thin gold film). The optimal parameters of the donor films (thickness ~ 40 nm) ensured the formation of single nanoparticles with a diameter $80-90$ nm with a high degree of control. The particles were transferred using a laser setup with the following parameters: $\lambda = 515$ nm, $\tau = 300$ fs, $f = 10$ kHz, $E = 6$ nJ. LOMO 60X NA 0.85 lens was used in the experiments that allowed to focus the beam with a diameter of 1 μm by level $1/e^2$. The transfer process control system based on the dark field microscopy made it possible to effectively control the transfer process (control the period and size) of the formed structures within the dark field image. Due to the vacuum fixation of samples on the slide tables the mechanical impact on the donor-acceptor substrates was ruled out, which, in turn, eliminated the incorrect flatness and mutual misorientation of the samples relative to each other in height. The independent displacement of the donor-acceptor substrates made it possible to overcome the limitations associated with the frequency of structures formed, achieve reproducibility and stability of the process, and form the ordered structures from groups of materials in a single technological process.

The developed method expands the options of laser microfabrication of meta-surfaces and opens up the prospects for fabrication of integrated devices with a nanometer precision.

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Conflict of interest

The authors declare that they have no conflict of interest.

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