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A versatile technology for colloidal crystal transfer using parylene coatings and hydrosoluble polymers

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Abstract. We propose a novel versatile colloidal crystal transfer technique compatible with a wide range of substrates regardless of their size, material, and wettability. There are no inherent limitations of colloidal particles material and size. The method possibilities are demonstrated via the colloidal transfer on quartz, glass substrates with a flat and curved surface, and the fabrication of 3D colloidal structure with 5 overlaid colloidal monolayers. The process occurs at a room temperature in water and is independent from the illumination conditions, which makes it ideal for experimental manipulations with sensitive functional substrates. We performed the nanosphere photolithography process on a photosensitive substrate with a transferred colloidal monolayer. The metallized hexagonal arrays of nanopores demonstrated a clear resonant plasmonic behavior. We believe that due to its high integration possibilities the proposed transfer technique will find applications in large-area surface nanotexturing, plasmonics, and will speed up a device fabrication process.

1. Introduction

Colloidal nanoparticles, arranged into hexagonal close-packed structures and often referred as colloidal crystals, have been a subject of intense research for decades due to their capabilities for light-matter interactions [1]. The interest is caused by their prospective use in a photonic crystal regime [2], Purcell factor enhancement [3], structure color generation [4, 5] inspired by natural opals. Along with 3-dimensional multilayered colloidal crystals, colloidal monolayers are a flexible platform for resonant and non-resonant optical effects: moth-eye mimicking structures [6, 7, 8], photovoltaics and light harvesting [9], Raman spectroscopy substrates [10], optical sensors [11].

The colloidal crystal deposition is an important part of device fabrication as it determines the close-packed structure quality and, consequently, its diffraction

properties. A variety of methods have been proposed, such as spin-coating [12], dip-coating [13], electrophoretic deposition [14] and colloidal self-assembly at air-liquid interface (Langmuir-Blodgett technique) [15]; they can be called "first principle" methods in analogy with first principle simulations, because they explicitly treat single nanoparticles as building blocks of the future colloidal crystal, study forces between them and substrate [16], study the influence of experimental parameters on the colloidal crystal quality [17, 18]. Therefore, the first principle methods are important from the fundamental point of view, and allow producing high-quality colloidal crystals as well.

However, the methods introduced above usually consider a certain combination of substrate and/or nanoparticle materials, thus limiting the applicability of elaborated experimental conditions to the most common cases only, like, for example, silicon/glass substrate and silica/polystyrene nanoparticles [14, 15]. In order to expand the applicability of self-assembled colloidal crystals, a relatively new branch of methods has been developed based on a colloidal crystal transfer from initial substrate to the final one [19, 20, 21, 22, 23, 24]. In contrast to first principle methods, these techniques do not generally perform a colloidal self-assembly as the most sophisticated and delicate procedure, but are aimed for the transfer of already prepared colloidal crystals on device being fabricated.

Authors of [20] describe a particle transfer printing with a thin water film as an intermediate adhesion layer in a transfer process. The technology allows the selective transfer of colloidal structure to form complex 2D patterns, but is limited to silica spheres and silicon target substrates (with a thin oxide layer) only. In [23] a freestanding poly(vinyl alcohol) (PVA)-based colloidal films were proposed to use in photovoltaics, but the process simplicity was achieved at a cost of increased film thickness of 50-70 microns. A big thickness might be undesirable for applications where near-field colloidal-structure-induced diffraction effects (i.e. photonic nanojets) should be located in the vicinity of the target surface. The work [21] presents an elaborated approach for layer-by-layer transfer of colloidal crystals with an arbitrary controlled number of layers. The method demonstrates a good repeatability, allows composition of layers with different morphology, particle size and materials. On the other hand, this approach is difficult to implement into device fabrication with sensitive functional films demanding to the environmental conditions like photoresists and photocatalytic surfaces, because it utilizes tough steps like heating at 100 °C for 3h and PVA calcination at 550 °C for 5h. The same remark applies to the work [24] as well, where the sacrificial polymethylmethacrylate (PMMA) thin film was degraded under 254 nm UV irradiation for 30 min. It should be noted that intensive UV irradiation during the transfer process is incompatible with perspective Nanosphere Photolithography (NPL) method of surface nanostructuration [25, 26], where photonic nanojets are used for resist exposure. NPL-fabricated metasurfaces are used in active photonics [27], surface texturing [28], plasmonic sensors [29] etc.

We believe that the progress in nanofabrication will definitely require the colloidal crystal depositions on non-conventional substrates with complex geometries and physical

properties – curved, large-area hydrophilic/hydrophobic functional surfaces – which are demanding about environmental and treatment conditions. Consequently, standard self-assembly and transfer techniques are often not applicable. In this paper, we propose a novel technique of colloidal crystal transfer that is free of the constraints mentioned above. Our approach can be used for the deposition of inorganic (glass, ceramic, ceramic oxides such as SiO_2 , Al_2O_3 , metallic) and organic (e.g., polystyrene) nanoparticles on any textured/non textured functional target surface regardless of its hydrophobicity and curvature, with only condition of being water-insoluble; the transfer process can occur at room temperatures and doesn't utilize UV irradiation, which is exceptionally important for photosensible substrates. The proposed technology is described in detail in the patent [30]. We demonstrate the colloidal crystal transfer on target substrates made of glass and quartz, flat and curved, with diameter of nanoparticles of 580 nm, 1.1 μm and 2.5 μm . Moreover, by repeating transfer steps colloidal multilayered structures can be obtained, we demonstrate up to 5 deposited layers. We integrated the transfer process into NPL routine and got arrays of hexagonally arranged nanopores of adjustable depth. The quality of the overall process was confirmed by observing the plasmonic-mediated Extraordinary Optical Transmission (EOT) through metallized structures.

Methods

Colloidal crystal transfer

Here we explain the principles of the proposed colloidal crystal transfer technique. Main steps are shown in Fig.1 and demonstrate two main procedures: the preparation of large-area thin sheets of colloidal crystal monolayers encapsulated between initial substrate and cover for the ease of transportation (Figs.1a-c) and the transfer process itself on any substrate on demand (Figs.1d-f).

The close-packed colloidal monolayer is formed on the initial substrate using conventional Langmuir-Blodgett technique [1]; without loss of generality silica particles were used in our study, whereas other materials are also possible due to the proper adaptation of Langmuir-Blodgett technique and experimental conditions. The size of colloidal particles used is 580 nm, 1.1 μm and 2.5 μm in diameter. The initial substrates were chosen to be 160 μm -thick polycarbonate sheets of size of 10×10 cm, they were fully covered with close-packed colloidal monolayer. In the second step an ultra-thin (40 nm) parylene coating is deposited on the top of colloidal crystal, denoted in green in Fig. 1b. Parylene polymer coatings have good chemical barrier properties, are thermally- and UV-stable for irradiation doses used in lithography [31], which makes these coatings compatible with NPL. The parylene mechanical stability and insolubility at room temperatures mechanically protects the colloidal crystal during all subsequent manipulations. In the third step (Fig.1c) the preparation of transportable colloidal-containing sheets is finalized by deposition of an initial 20 μm -thick hydrosoluble PVA cover via spin-coating of its 5 wt % aqueous solution. The resulting sheets with

Figure 1. The scheme of proposed colloidal crystal transfer process. a) The colloidal self-assembly and deposition on an initial substrate, for example, polycarbonate; b) Thin (~ 40 nm) parylene coating deposition, depicted as green transparent film; c) Deposition of an initial cover containing hydrosoluble polymer, for example, PVA; d) The colloidal monolayer and initial cover peeling off from the initial substrate; e) Contact with a final substrate; f) Dissolution of the initial cover in water.

encapsulated colloidal monolayers can be stored and transported without any special vibrational and temperature requirements, they only should be contained in standard office punched pockets to avoid a high air humidity.

The second procedure is the colloidal crystal transfer from the prepared sheets to any substrate on demand. As the adhesion of parylene-covered colloidal crystals with the initial cover made of PVA is much stronger than that with the initial polycarbonate substrate, the nanoparticles will stay on the lower side of the initial cover after a simple manual peeling (see Fig.1d and Fig.2a). The free-standing PVA-supported colloidal crystal is manually applied to the target substrate (Fig.1e and Fig.2b) with a certain pressure. As the target substrate can be of different materials, in order to increase the adhesion with it the sample is immersed in water for a short time ~ 1 s at room temperature, to provoke the PVA layer partial melting. Then, to delete water and harden the PVA layer again, sample is dried under a nitrogen stream, or can be placed on a hot plate at 30°C for delicate drying (Fig.2c). In order to complete the transfer the initial PVA cover should be eliminated. To do this, the sample from Fig.1e is immersed in a deionized (DI) water tank at room temperature for ~ 30 min, with subsequent water pumping out of the tank at low speed to ensure a high-quality colloidal transfer (Fig.2d). As a result, the close-packed colloidal crystal encapsulated in a polymer sheet is transferred on the target substrate (Fig.1f and Fig.2e).

Figure 2. a)-e) Photographs of the process of colloidal crystal transfer from initial polymer sheets to the target substrate (BK7 glass slides covered with photoresist Shipley S1805 in this case): a) the peeling off the initial cover with colloidal crystal; b) the initial cover with colloidal crystal mechanically applied to the target substrate. Scale bars in a) and b) denote 1 cm; c) samples after a short ~ 1 s immersion in water for partial PVA melting, dried under the nitrogen stream; d) the process of PVA dissolution in a water tank with samples oriented vertically. Water is pumped out of the tank at low speed via the tube, so only the parylene-covered colloidal crystal stays on the substrate. f) The photograph of nanopore arrays fabricated in the photoresist via NPL, using the transferred colloidal crystal as a nanojet-forming mask. Different zones on every sample in f) correspond to different UV exposure time. The diameter of transferred colloidal particles in a)-e) and the inter-pore distance in f) is 580 nm.

Nanosphere Photolithography

The Nanosphere Photolithography (NPL) is a high-throughput, relatively inexpensive approach for large-area surface nanopatterning, based on the inhomogeneous exposure of photoresist by nanojets [25]. The principle of NPL is sketched in Fig.3. The colloidal crystal monolayer transferred on the photoresist surface is UV-exposed during the time t_{exp} ; during the exposure every nanoparticle acts as a microlens, generates the nanojet and exposes the resist inhomogeneously [26]. We use a positive photoresist Shipley S1805 and a portable UV lamp E2107 2×6 W for UV irradiation at 254 nm (Fig.3a). It should be noted that, although the parylene film absorbs at wavelengths below 290 nm [32, 33], its thickness (40 nm) is not enough to affect the NPL process; practically it can lead only to a small increase of t_{exp} . As a substrate we used BK7 glass slides 3.7×2.5 cm, cleaned in a three-step wet-bench procedure (ultrasonic acetone bath, ethanol bath and

Figure 3. The scheme of NPL method. a) UV-exposure of the close-packed colloidal monolayer (with a 40-nm thin parylene film denoted in green) transferred on the photoresist surface; b) The removal of colloids in an ultrasonic bath; c) The formation of hexagonal arrays of nanopores during the resist development.

deionized water bath). The 600-nm thick resist layer was spin-coated on BK7 slides and soft-baked at 60 °C for 1 min on a hot plate. In the second step (Fig.3b) the colloidal mask is removed via the ultrasonic bath, and in the final step (Fig.3c) the sample is developed in MF-319 at 8 °C during the time t_{dev} . The developer dissolves the resist inhomogeneously, so the arrays of nanopores appear on the surface which repeat the hexagonal arrangement of initially transferred nanoparticles. The nanopore depth depends on values of t_{exp} and t_{dev} for a given photoresist and developer.

Samples characterization

The quality of colloidal crystal transfer was characterized using scanning electron microscope Nova NanoSEM 200 FEI; the nanopore depth was measured with atomic force microscope Bruker Dimension XR; the transmission spectra were obtained via UV-Vis-NIR spectrophotometer Agilent Cary 5000.

Results and discussion

Transfer on different substrates

The colloidal transfer in Figs.2b-e and NPL process in Fig.2f performed for two samples at once demonstrates a high-throughput, repeatability and stability of the method. The colloidal crystal can be transferred on target substrates of different materials and geometries, as demonstrated in Fig.4: quartz, glass substrates with plane and curved geometries. Moreover, consecutive transfer of monolayers on the same substrate allows creating 3D colloidal structures with controlled number of layers: sample in Fig.4d consists of 5 colloidal layers on its right side (the silica nanoparticle diameter is 580 nm in this case). The length of transferred colloidal stripes here was specially chosen to be different to reveal the high quality of every transferred layer. It should be noted

Figure 4. Photographs of colloidal crystals transferred on different target substrates: a) monolayer on quartz; b) monolayer on BK7 cylinder; c) monolayer on BK7 glass slide, covered with S1805 photoresist; d) from 1 (on the right) to 5 (on the left) monolayers on a polycarbonate substrate. Scale bar in a) is the same for all photographs and denotes 1 cm. The diameter of colloidal particles used for the transfer is 580 nm.

that in all samples in Figs.2 and Figs.4 the inner parts of colloidal layers are practically free of defects visible by naked eye. The mechanical defects are concentrated mostly on the layers edge and can be of two types, as shown in Fig.4d insets: the layer rupture because of air bubbles trapped between the substrate and colloidal film (upper inset) and colloidal film folds which appear when the edge of a colloidal film detaches from the substrate during the water pumping out of the tank (lower inset). The both types of defects are avoidable with further improvements of the transfer process routine.

Figures 5b-f show the SEM photographs of the top surface of the sample from Fig.4d (the same as in Fig.5a) at regions with 1-5 transferred colloidal monolayers, respectively. The quality of hexagonal polycrystalline packaging does not depend on the layer's number and is possible due to the mechanical support of the thin parylene film.

Nanopore arrays fabricated via NPL

Due to the moderate experimental conditions under which the proposed transfer method occurs (room temperature, independence from the room illumination, water-assisted transfer), functional substrates conserve their properties. Consequently, substrates with transferred colloidal crystals can be used in photonic devices or be integrated into advanced experimental manipulations. Here we use the Nanosphere Photolithography (NPL) as an example of such an advanced fabrication procedure.

Figure 6a shows the same two samples as in Fig.2f with only one colloidal layer (nanoparticle diameter 580 nm). Every sample has separate diffractive areas (8 areas

Figure 5. a) Photograph of the sample with up to 5 colloidal monolayers (the same as in Fig.4d); b)-f) SEM photographs of sample surface at regions with 1-5 transferred monolayers, respectively. Scale bar in b) is the same for all photographs b)-f) and denotes 5 μm . The diameter of colloidal particles is 580 nm.

Figure 6. a) Photograph of two samples with nanopore arrays, fabricated via NPL from transferred colloidal monolayers (the same as in Fig.2f). White arrows indicate the directions of nanopore depth growth. The diameter of initially transferred colloidal particles was 580 nm; b) AFM measurements of nanopore depths in every UV-exposed area of Samples 1 and 2. Every area was exposed during the time t_{exp} ; c), d) and e) AFM-measured topographies of Sample 2 areas with $t_{exp} = 15$ s, 30 s and 45 s, correspondingly; f) and g) AFM-measured topographies of NPL-fabricated nanopore arrays, obtained from the transfer of colloidal particles of diameters 1100 nm and 2500 nm, respectively. Nanopore depths for f) and g) are 116 nm and 200 nm, respectively.

for Sample 1 and 7 areas for Sample 2), which were nanotextured via NPL with different UV-exposure times t_{exp} , and which consist of hexagonally arranged nanopores. The development time for all areas of both samples is the same $t_{dev} = 4$ s. The nanopore depth increases with t_{exp} , white arrows in Fig.6a indicate directions of the nanopore depth growth. The experimentally measured dependence of grating depth over the exposure time t_{exp} is shown in Fig.6b for Samples 1 and 2. The fact that both curves

almost coincide with each other proves the repeatability of results. The AFM-measured topographies of nanopore arrays are shown in Figs.6c-g (Fig.6f for nanoparticle diameter 1100 nm, Fig.6g for 2500 nm, other images for 580 nm), they repeat the polycrystalline arrangement of initially transferred colloidal monolayers.

Experimental measurements of EOT

We utilized NPL to demonstrate the processing possibilities of the proposed colloidal transfer technology. Due to the NPL-compatibility the transferred thin films can be employed in active photonics [27], surface texturing [28], plasmonic sensors [29] etc. In this section we demonstrate that resonant plasmonic excitations are possible in metallized nanopore arrays, prepared via NPL and colloidal transfer, despite the disordered polycrystalline nature of samples. These results are consistent with our previous theoretical and experimental studies [26, 34].

For experiments we chose a sample analogous to one from Fig.4c with NPL-fabricated array of nanopores from previously transferred close-packed colloidal particles with diameters of 580 nm. The NPL exposure time is $t_{exp} = 10$ s, resist development time is $t_{dev} = 4$ s, and AFM-measured nanopore depth is 40 nm. The resist layer thickness is 600 nm. In order to get a plasmonic structure the nanotextured resist surface was metallized with a 50 nm-thin gold film using a thermal evaporation in vacuum, and the second 600 nm-thin resist layer was spin-coated on the top. Thus, the symmetrical "Insulator-Metal-Insulator" (IMI) geometry was achieved, which supports the plasmonic Extraordinary Optical Transmission (EOT) effect [26, 35].

Figures 7a,e show the measured transmission spectra of the prepared structure in TM and TE polarizations, respectively, for incidence angles 0° , 5° and 10° . At normal incidence both spectra in Figs.7a,e demonstrate almost identical plasmonic peaks at $\lambda_2 \approx 840$ nm; this resonant wavelength for the diffraction order (m, n) can be estimated by the equation

$$\lambda_{EOT} = \frac{\sigma_0}{\sqrt{\frac{4}{3}(m^2 + mn + n^2)}} \sqrt{\frac{\varepsilon_d \cdot \text{Re}(\varepsilon_m)}{\varepsilon_d + \text{Re}(\varepsilon_m)}} \quad (1)$$

for the hexagonal lattice [36, 26]; ε_d and ε_m are dielectric permittivities of dielectric and metallic layers, respectively, and σ_0 is the interpore distance defined by the diameter of the self-assembled particles. Taking $\varepsilon_d \approx 1.6^2$ for the S1805 dielectric resist, $\varepsilon_m \approx (0.16 + 5.3i)^2$ for gold and $\sigma_0 = 580$ nm, we estimate the EOT wavelength $\lambda_{EOT} \approx 840$ nm defined by 6 symmetrical first diffraction orders $(m, n) = (\pm 1, 0), (0, \pm 1), (\pm 1, \mp 1)$. It is worth mentioning that, in contrast to ideally periodical 2D hexagonal lattices with 6 discrete first diffraction orders (Fourier harmonics), the studied polycrystalline structure possesses a blurred diffraction ring because of randomly oriented nanopore crystallites of limited size [26].

With the increase of incidence angles the EOT spectral behavior becomes different in TE and TM: the EOT peak splits in TM, but stays at the same position λ_2 in TE. It

can be explained using the Ewald's sphere approach in relation to plasmonic excitations, described in detail, for example, in [37].

Figures 7b-d explain the appearance of two EOT peaks for TM polarization in polycrystalline disordered structure at wavelengths λ_1 and λ_3 by multiple intersections of the plasmonic circle (denoted in black) with a blurred grating-imposed first order diffraction ring (denoted in green). These multiple intersections define multiple surface plasmon propagation directions, which merge into broad sectors (defined in brown). The width of sectors increases with the decrease of nanopore grain size. Thus, at inclined incidence there are two ways for plasmonic excitations in TM, shown in Figs.7b,d, and consequently two broad EOT peaks at λ_1 and λ_3 .

The coupling of TE-polarized light to plasmons at λ_1 and λ_3 is suppressed, because propagation sectors are almost perpendicular to the light polarization at these wavelengths. However, the coupling is still possible at λ_2 , as shown in Fig.7g.

Figure 7. Extraordinary optical transmission through polycrystalline arrays of nanopores, fabricated via NPL and colloidal crystal transfer. a) Transmission in TM polarization; b)-d) diagrams in reciprocal space for different incident wavelengths denoted above each diagram. Black circles are plasmonic spheres with plasmon resonant wavevectors, green circles show first Fourier harmonics of polycrystalline 2D gratings (first diffraction orders). Colored brown sectors inside plasmonic spheres denote sets of wavevectors at which plasmonic resonances occur. Yellow arrows inside colored sectors demonstrate the incident light polarization; e) Transmission in TE polarization; f)-h) the diagrams similar to ones in b)-d), for plasmonic excitations at TE polarization.

The clear experimental observation of EOT in samples, fabricated via the proposed colloidal transfer method, confirms the preservation of diffraction structure quality

during this transfer and demonstrates the possibility of its integration into multi-step fabrication processes.

Conclusions

In conclusion, we proposed a novel versatile colloidal crystal transfer technique compatible with a wide range of substrates regardless of their size, material, and wettability. There are no inherent limitations of colloidal particles material and size, too. In order to demonstrate the method possibilities we performed the colloidal transfer on quartz, glass substrates with a flat and curved surface, and prepared 3D colloidal structure with 5 overlaid monolayers. In addition, the process occurs at a room temperature in water and is independent from the illumination conditions. Consequently, it does not damage sensitive functional substrates and therefore can be implemented into more sophisticated fabrication chains.

As an example of functional substrates the photoresist S1805 was chosen; we managed to use the transferred colloidal monolayer as a mask for nanosphere photolithography. The metallized hexagonal arrays of nanopores demonstrated a clear extraordinary optical transmission behavior.

We believe that due to its high integration possibilities the proposed transfer technique will find applications in large-area surface nanotexturing, plasmonics, and will speed up a device fabrication process.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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