A set of multifunctional equipment for deposition of colloidal photonic crystal films and monolayers

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Abstract. It is impossible to obtain colloidal photonic crystal film of given properties without special equipment. In order to minimize the influence of external disturbing influences such devices should be automated. To obtain large-area or hetero structures it is rational to combine in one universal device equipment for the implementation of several depositional methods. In this paper the authors present two automated devices for both special and universal purposes. They can be used to obtain films and monolayers by vertical lifting of the substrate from the colloidal suspension, vertical deposition by pumping out the suspension, electrophoresis and Langmuir-Blodgett technique and its combinations.

1. Introduction
The colloidal photonic crystal (PC) film deposition technologies are well developed under laboratory conditions and their applications are presented in publications by many authors [1-4]. Colloidal films are obtained by the methods of natural sedimentation, vertical lifting of the substrate from the suspension (solution), vertical deposition by evaporation or pumping out the suspension, electrophoresis, Langmuir-Blodgett technique and centrifugation [5]. However, the transition to the production of industrial samples is hindered by the low reproducibility of processes. This is especially critical when depositing large area films. The use of automated systems can significantly improve reproducibility [6, 7]. Previously, the authors presented their equipment and its capabilities [8, 9]. The interest in PC multilayer heterostructures and structures with point or line defects necessitates the possibility of combining several methods of film deposition in one unit for their simultaneous or sequential use, and realizing the possibility of the defect formation in the deposited structure. Equipment automation eliminates the interaction of factors, the influence of dominant factors and the time factor. This paper presents two units. One of them implements the vertical lifting method and is designed to produce planar structures of a given thickness with the possibility of forming linear defects using a drive that intermittently moves the substrate. The other is intended for the deposition of films and monolayers by vertical deposition, electrophoresis and Langmuir-Blodgett methods and their combinations.

2. Equipment and methods
When producing films by vertical deposition (VD) the substrate is placed vertically in the colloidal suspension and is gradually exposed by evaporation or other slow solvent removal or substrate lifting. The vertical lifting (VL) device is designed for the deposition of reproducible large area PC colloidal films. This method is one of the easiest to implement, and at the same time it is sufficiently technologically advanced in order to provide the variability of deposition modes. The device consists
of three dismountable parts. The upper part is made of metal sheets 2 mm thick with holes for display, keyboard and power supply system outputs, and contains all the components of the automated control system (ACS) inside. The middle part is a working area of the device, made of organic glass to simplify observation of the process from outside. It also has additional metal frames installed in the corners, to increase the strength of the structure and avoid cracking of organic glass. The lower part is a massive frame that acts as passive vibration isolation. Besides, in case of modernization of the device, a cavity is provided in the lower part for installing a thermal heating element, if there’ll be a need to heat the solution during the deposition process. Figure 1a shows a device diagram.

![Diagram of the device](image)

Figure 1. 3D model of: (a) VL device, (b) universal device.

Power supply of the device elements is carried out using a pulsed power source 220 V - 12 V AC-DC transformer. The linear motion of the substrate is caused by the transformation of the rotational motion of the electric motor operating from a voltage of 12 V, having an output shaft diameter of 6 mm and a nominal rotation speed of 3 RPM. Motor speed, direction of rotation and braking functions are controlled by a motor driver that maintains 12 V output channels. Also, a flywheel of greater mass and radius is installed on the engine output shaft to reduce inertia during acceleration and deceleration. This type of construction allows smoother pulling and avoids the appearance of irregularities on the substrate surface. The ACS system is an Arduino microcontroller that controls the electric motor driver according to the wide pulse modulation (PWM) principle. It also has a 3.2-inch screen connected to it to display the status of the application process and to simplify data processing by the user. Data processing can be performed both from a personal computer and directly through the keyboard built into the case. The control system in combination with the lifting mechanism allows to achieve the lifting speeds we need in the range from 0.01 mm/min…3 mm/min at maximum smoothness. Speed control problem is solved by installing two single-pass (cross-beam) photoelectric sensors. The first of them is located directly on the output shaft of the electric motor, to ensure accurate control of the rotation speed, and to identify problems associated with its wear or inaccurate tuning of the control system. The second one is located as close as possible to the substrate holder, which makes it possible to take into account all errors that accumulate in the system and show the actual lifting speed. One of the important factors is vibration damping during deposition, therefore the lower part of the device is made of a 20 kg metal plate, which is many times greater than the weight of the rest of the structure. And also, the rotation of the engine is transmitted to an inextensible tape, to which a substrate holder and two weights with a total mass of 2 kg are attached, which makes it
possible to minimize the vibrations transmitted from the rotation mechanism to the substrate pulled out of the solution.

The universal device is designed to implement deposition of reproducible colloidal films by methods of VD, electrophoresis (E), movable meniscus (MM), Langmuir-Blodgett (LB). Due to the semiempirical analysis was defined a spectrum of input factors influencing on the process and parameters of the film: solution temperature (VD, MM), environmental conditions (VD, MM, LB), pH of the solution (E), substrates potential (E), pumping rate (VD, MM, LB). To improve the quality and accuracy of the films parameters, it is necessary to provide high-precision control of the above-described input parameters. The automation of the device allows to minimize an error produced by the operator in case of inaccurate adjustment. Combination of different methods in one unit allows obtaining films with fine-controlled repeatable parameters due to combined impact of different factors.

Figure 1b shows a universal device diagram. Bath is equipped with the module for stirring and regulating the temperature of the solution. The device consists of easily replaceable modules and provides the ability to control the input parameters and to improve the quality of the films. The temperature control system consists of a 100W ceramic heater and a temperature sensor with a measurement error of 0.5 °C. This system is intended mainly for carrying out the MM and VD processes. By increasing temperature can be achieved lower viscosity of the solution and better transfer of particles to the deposition zone. Control unit is also equipped with a temperature-humidity-pressure sensor, which allows measuring atmospheric pressure with an error of 1 Pa, relative humidity with an error of 3 %, and ambient temperature with an error of 1 °C. These input factors mainly affect the VD, MM and LB processes, since evaporation rate of the solution depends on the described above parameters. As a result the solution level will descend, regardless of whether pumping occurs. The pH of the suspension affects the double electrical layer (DEL) of the particles. At low thickness of the DEL may occur coagulation of colloidal particles in solution due to the absence of a repulsive charge, which significantly reduces the quality of the films. Also, this factor affects the E process, since it directly affects the force acting on the particles in the electric field. Equipped pH control sensor has an error of 0.1 pH. Control of substrate potential is carried out using the adjustable DC-DC voltage converter and digital potentiometers, adjustment range is 0 to 24 V automatically, while the adjustment error is 5 mV. It is able to adjust the current up to 5 A. The voltage sensor has an error of 1 mV. Pumping of the suspension is carried out using a peristaltic pump, which is safe for particles from damage during the pumping process. The pump is driven by a stepper motor with a driver at microstepping mode 1/256 to ensure smooth pumping. Pumping speed adjustment rate is 0.1 rpm to 100 rpm. Minimal descend speed on used bath is 0.3 mm/min. The stirring module is necessary to prevent the coagulation of particles in the suspension and for their redistribution over the volume, which leads to the production of films of the best quality. Also, this module is useful while heating the solution, accelerating the temperature equalization in the bath during operation. The control unit also equipped with data recording system. User is able to connect to the installation from PC via Bluetooth module for further data analysis in developed software. It is also applicable while setting the process parameters, process may be controlled directly by PC/laptop/tablet.

3. Results and discussion
Both projects have been implemented and are used in the laboratory practice of students. Figure 2 shows photos of devices, AFM images, and spectra of colloidal films obtained in the course of routine experiments during the educational process.
When testing the functionality of the equipment, we obtained monolayers and films of colloidal polystyrene (PS) particles with a COOH group and got good reproducibility of the position of the photonic band gap - the error is about 2...5 nm for standard samples of polystyrene particles with a diameter of 200 nm ... 500 nm.

4. Conclusion
This work shows that automation of equipment for obtaining colloidal films makes it possible to deposit PC films of acceptable quality even in routine research. Since most of the process parameters are well controlled and the influence of random factors is minimized, devices can be used for large-scale scientific research. The results of this work will be used for the controlled synthesis of nanoperiodic homo and heterostructures and monolayers for optoelectronic devices.

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