



Robust surface patterning by parylene-reinforced breath figures: An enabling tool for liquid crystal microcell arrays

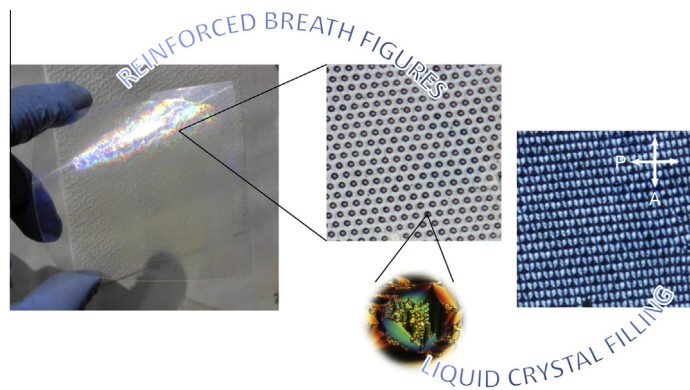


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GRAPHICAL ABSTRACT



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ABSTRACT

We propose a novel fabrication method for realizing robust and solvent resistant honeycomb films by combining parylene deposition with the polymer auto-organization phenomenon leading to breath figures. Parylene CVD process is suitable for producing a conformal protective layer on the honeycomb surface, reinforcing and stabilizing this structure. We demonstrate that the stabilized porous films can be filled with chloroform solution of fluorescent materials, without losing the integrity of their microstructure. In addition, liquid crystals can be encapsulated inside the patterned surface between two layers of parylene, showing interesting organization features dictated by the spatial constraints. This is the first reported application of breath figures with liquid crystals, suggesting a new approach for the development of honeycomb-based liquid crystal cells for flexible displays.

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1. Introduction

Self-assembly has emerged as one of the most significant paradigms for the fabrication of new materials of technological interest due to its advanced capability for generating complex nano and

microstructures [1]. The possible structures encompass a wide range of morphologies emerging from microphase separation of block copolymers [2], dewetting [3], coffee rings [4], crystallization of colloids [5], and supramolecular organization [6] among others. In this context, the breath figures (BF) approach represents an attractive and practical tool for the nano and micropatterning of thin polymer films [7]. In fact, the simple drop or spin-casting of a polymer solution under a controlled humidity environment

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allows for the realization of highly ordered and tunable honeycomb structures, which have lately been proposed for a plethora of different applications [8–11].

Despite the simplicity and fast realization of this technique, a main drawback of BF films is their intrinsic fragility. In fact, the walls separating the voids in a typical BF layer are only a few nanometers thick, which greatly limits their robustness. The most convenient materials suitable for the preparation of BFs are either commercial or customized polymers such as polystyrenes and polyacrylates, hence their patterned thin films swell or dissolve very quickly in most of organic solvents. In the last years, researchers have proposed different methods to increase the robustness of BF films, including polymer stabilization by UV crosslinking [12–14] and plasma CF_4 treatment [15]. A more delicate approach consists in blending the polymer solution with a colloidal dispersion of inorganic particles, so that the resulting honeycomb structure is maintained by the inorganic skeleton even after removal of the

organic part by calcination [16]. All these procedures are, anyway, only partial and incomplete solutions to the problem, especially in the view of possible application of the microstructures in functional devices. UV and plasma stabilization never lead to complete crosslinking, and, most of the times, the stabilization is sufficient to preserve the structure after a quick contact with the organic solvent but not for prolonged wetting or soaking, while calcination requires high temperatures that restrict the range of functional materials to the only inorganic ones.

An alternative approach for the stabilization of BFs is to cover them with a thin coating of a solvent resistant material. Poly(*p*-xylylene) (trade name: parylene) is widely used as conformal coating for the protection of devices, components, and surfaces, in many different applications [17,18]. It is a chemically and biologically inert and stable material, with good transparency, fair barrier capability and electrical insulation properties, and insolubility in all organic solvents [19]. Because in the parylene CVD process, the polymer is formed directly on the surface that has to be coated at room temperature, it is suitable for being applied on BF films.

The work herein details the preparation of robust honeycomb films by means of BF templating and parylene deposition. The conformal parylene coating stabilizes the honeycomb films, making these microstructures suitable for the further filling with an organic solvent solution (fluorescent dyes, liquid crystals (LCs)), thus paving the way to the development of new functional materials with enhanced technological interest.

2. Experimental

Two kinds of BF arrays were employed in this study: close-packed randomly distributed large pores and close-packed highly ordered small pores. For the preparation of the large ($\approx 10 \mu\text{m}$) scarcely ordered microporous templates, CS_2 solutions of a commercially available polystyrene purchased from Aldrich (M_w 230 kDa, 18 mg/mL) mixed with 1% (w/w) of AliquatTM 336 (Aldrich) were employed. For the small ($\approx 3 \mu\text{m}$) and highly ordered microporous templates, we used CS_2 solutions of an amphiphilic polystyrene synthesized in our laboratory (M_w 84.7 kDa) [20]. Films were obtained by casting the proper volume of these CS_2 solutions on square pieces of PET foil ($6 \times 6 \text{ cm}^2$) under a flow of moist nitrogen (85% R.H. at 25°C), obtained by flowing nitrogen

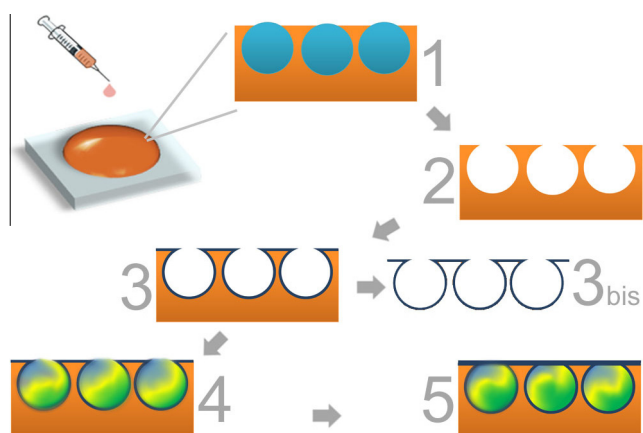


Fig. 1. Schematized fabrication route. A polymer solution is cast under humid atmosphere to induce the formation of breath figures (1). Water evaporates off leaving a porous imprint in the polymer film (2). A thin layer of parylene is deposited on it (3). The polymer underneath the parylene layer can be removed by solvent washing (3bis). The honeycomb film is filled with a liquid material (4). The liquid is encapsulated into microcells by depositing a second parylene layer on top (5).

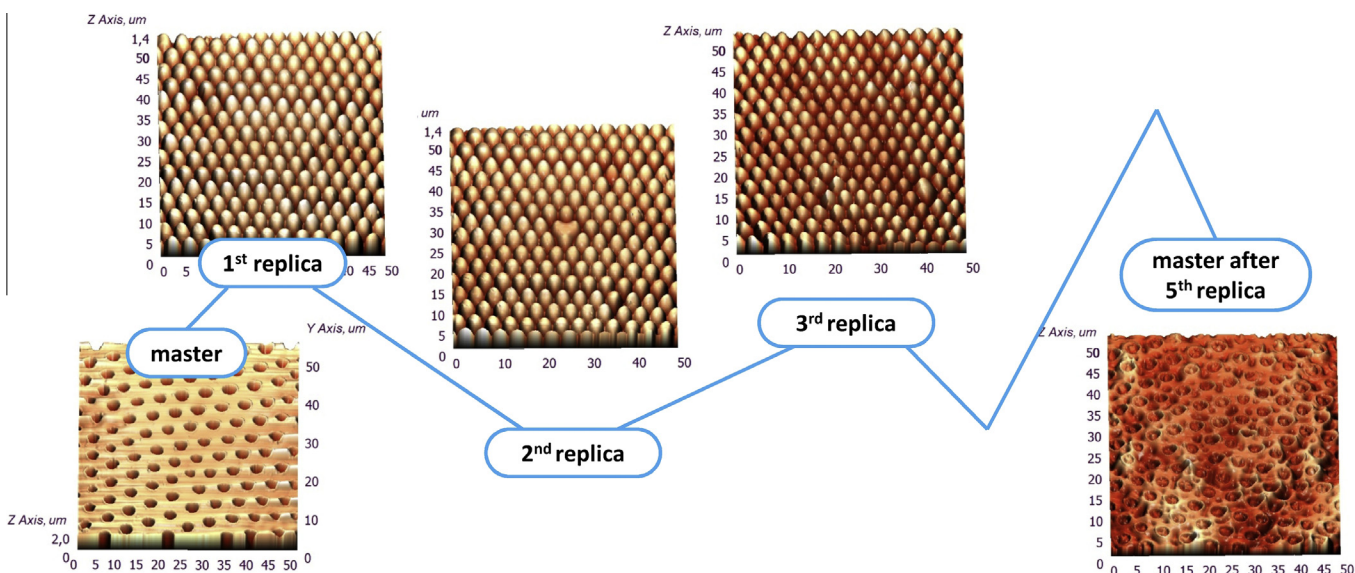


Fig. 2. AFM images of a polystyrene honeycomb master coated with parylene before and after five replica molding processes, and of the PDMS patterns resulting from the first three processes.

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