Glancing Angle Deposition Thin Film Microstructures for Microfluidic Applications

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Micro- and nanometer scale structures promise to be of great importance in the advancement of biotechnology. Recent studies have demonstrated the capabilities of these structures as artificial separation matrixes for microfluidic devices.[1] In comparison to conventional gels which have random pore size distribution, fabricated artificial gels have controllable and measurable porosity which make them easier to integrate with devices that sort, separate, and analyze molecules on the basis of size or mechanical properties. Fabricated artificial gels have been shown to provide high efficiency and resolution in the molecular separation of biomolecules such as DNA and proteins.[1−3] Most of the previous methods for fabricating artificial micro- and nanometer scale fluidic structures have involved top-down lithography and etching processes. As a result, most of the materials used to fabricate artificial fluidic structures have been glass and selected semiconductors limited to standard microfabrication technology. This, however, limits the study of artificial structures made of materials that would interact with individual molecules at the nanometer scale. The work reported here describes a new glancing angle deposition (GLAD) technique for engineering highly porous microstructures in a wide variety of materials and with potential use in microfluidic analysis.

The GLAD technique involves physical vapor deposition of thin films onto rotating substrates oriented at highly oblique angles relative to the incident vapor stream.[4,5] In the absence of significant diffusion, strong geometrical shadowing within the growing films leads to highly porous and columnar microstructures.[6] The film mean density (typically about 30% of the bulk) is given by the vapor incidence angle, while the shape of the microstructure is controlled by substrate rotation.[7] In a recent advancement of GLAD, based on decoupling of the microcolumn growth direction from the vapor arrival direction, control over the size and distribution of the pores in the film has been achieved.[8] The advanced GLAD film growth algorithm involves sweeping the horizontal vapor arrival direction from side to side about a central axis defining the direction of the microcolumn growth. This way, the arriving vapor can be forced to accumulate in a manner that matches the natural width of the thin film microstructures. On smooth areas of a substrate, this porosity engineering capability allows a fairly compact thin film morphology with pore sizes on the order of a few nanometers to be deposited. Meanwhile, on areas of the *same* substrate pre-patterned with a periodic array of small protrusions or seeds, designed to intercept vapor and enforce film column nucleation at predetermined locations, an open film morphology of periodic microcolumns with pore sizes on the order of hundreds of nanometers can be attained.[9] The selectivity in film porosity translates into a differential in the resistance to liquid flow, and with the pattern of protrusions arranged to form a microfluidic circuit design, this effect can be exploited to generate embedded flow channels within the GLAD film. The periodically arranged microcolumns inside the flow channels may act as a sieving matrix in molecular sorting and analysis, with spacing of the periodic microcolumns controlled via the lattice period of the array of protrusions. Thus, separation based on size exclusion chromatography (SEC) may also be achieved with this new GLAD method by fabricating a sieving matrix with narrow and wide gaps.

Figure 1 shows a schematic representation of a microfluidic GLAD film fabrication process. A soda-lime glass substrate was patterned with protrusions written in Clariant AZ-1518 UV resist using a Heidelberg Instruments DWL 200 laser direct write lithography system and developed with Microposit 354 developer. The seed lattices defining the flow channels had a tetrahedron geometry with a lattice period of 1 μm. The silicon GLAD film was deposited on the patterned glass substrate in a Kurt Lesker electron beam evaporation system at a pressure of 0.1 mPa and a deposition rate of 10 Å/s. During the deposition the substrate was tilted to an angle of 84° relative to the direction of the impinging vapor, and the substrate was rotated to create a helical microstructure. Figure 2A illustrates a microfluidic channel defined in a GLAD film. In those parts of the thin film deposited on the patterned protrusions, shown in detail in Figure 2B, relatively large gaps of 100−200 nm exist between the periodically arranged microcolumns. The pore size depends on the size of the seed used (varies from 100 nm to 2 μm wide) and the overall porosity of the film governed by the substrate tilt angle. Those parts of the film deposited on the bare substrate, as shown in Figure 2C, exhibit a film morphology that is much more compact, with a pore size less than 10 nm. This is the basis both for selective liquid flow inside the patterned channel and for potential SEC in the channel. The GLAD approach to the fabrication of microfluidic networks, thus, consists only of patterning of a protrusion array and of a single bottom-up thin film deposition step, which eliminates the tedious top-down processing required for traditional microfluidic archi-

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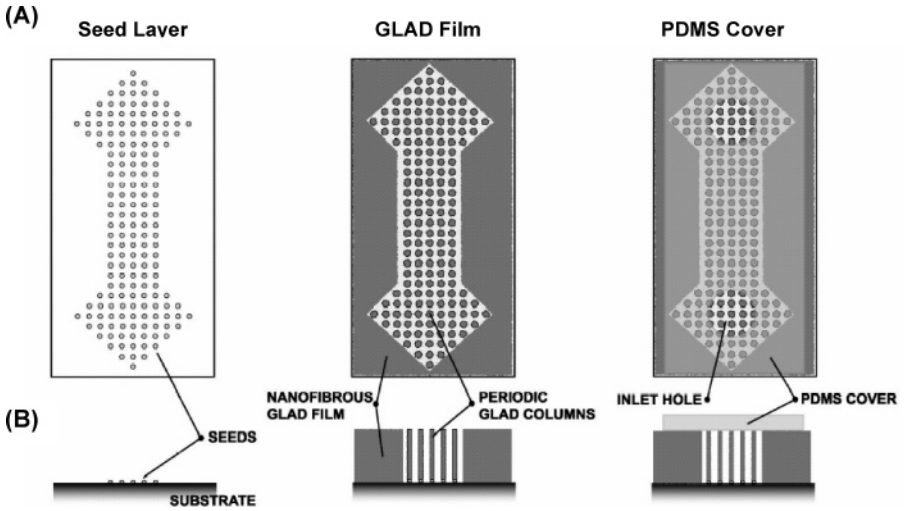

Figure 1. (A) Top view schematics illustrating the three steps in the fabrication of a microfluidic GLAD film. The substrate was patterned with an array of seeds which defined the flow channels. The GLAD film was then deposited resulting in periodic film structure on the patterned regions and a fairly compact morphology on the bare regions. A PDMS cover was used to seal the flow channels. (B) Cross-sectional view.

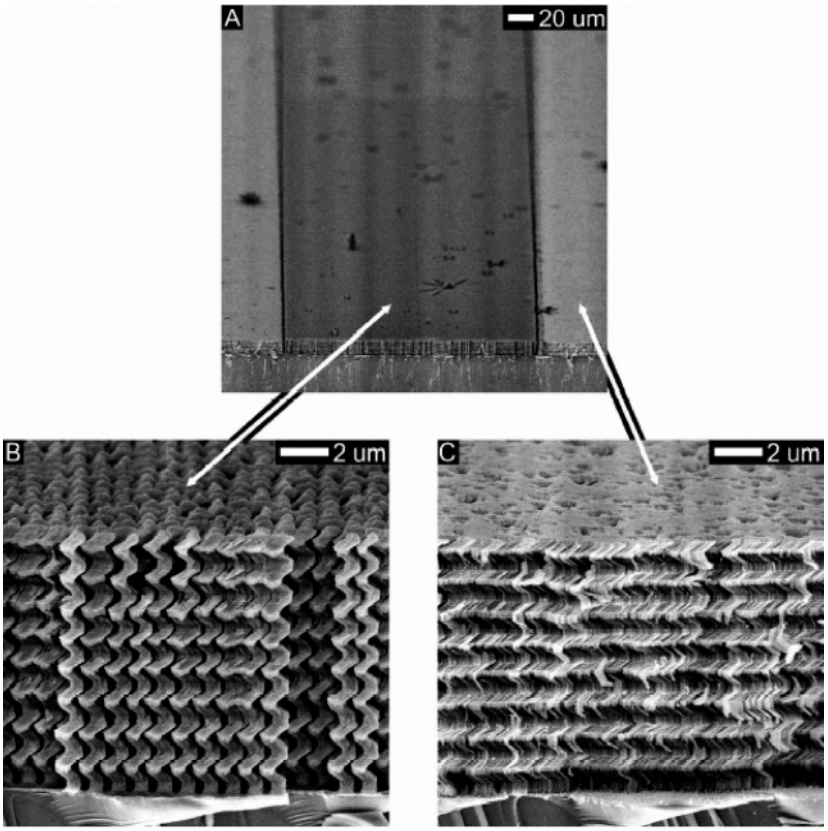

Figure 2. (A) Scanning electron microscopy image of a GLAD microfluidic channel, 200-μm wide. A higher magnification showing (B) inside the channel with large gaps between the microcolumns and (C) outside the channel with a fairly compact film morphology.

tectures. In principle all materials amenable to physical vapor deposition could be employed to making a GLAD-based microfluidic circuit. So far, films made of silicon, titanium, and titanium oxide have been fabricated with the new GLAD method. The surface chemistry of the GLAD films may also be tailored via chemical modification, which widens their potential analytical applications.[10]

Figure 3 shows a sequence of video frames illustrating the flow of liquid through a double-T microfluidic network fabricated using the new GLAD method. The flow channels appear as gray lines and have a periodic film structure identical to that shown in Figure 2b. The channel was 3-cm long and 200-μm wide. The regions outside the channel consist of an aperiodic film structure identical to that shown in Figure 2c and appear brown in color due to differences in the reflection of light. The thickness of the GLAD thin film was 10.5 μm. A self-sealing poly(dimethylsiloxane) (PDMS) elastomer layer with holes aligned with the inlet and outlet ports of the microfluidic circuit was employed as a cover. Figure 3A shows the microfluidic device before the

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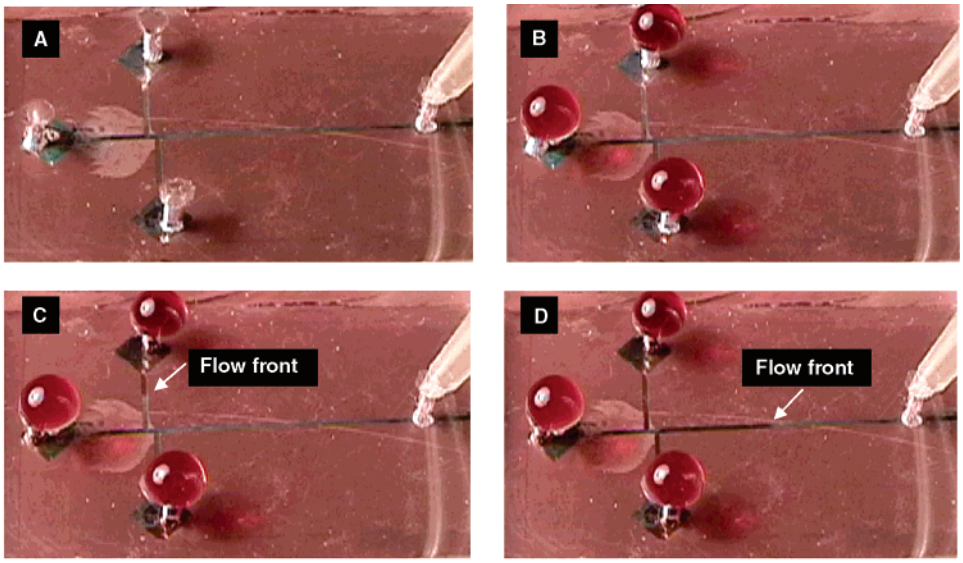

Figure 3. Sequence of video frames illustrating the flow of liquid through a microfluidic GLAD device. (A) Empty device, (B) inlet ports were loaded with a dye solution, (C) outlet vacuum was turned on and flow proceeded through the inlet ports and into the shorter side channels giving the periodic GLAD film microstructure a darker appearance due to wetting. Part D was obtained when the liquid flow front was at about the midpoint of the main channel.

liquid flow experiments. The flow experiments were carried out using a red-colored solution of a ruthenium dye dissolved in a 1:1 mixture of water and methanol. The red color of the dye solution was distinguishable from the light brown background of the GLAD films. Figure 3B shows the three loaded inlet ports while the fourth port at the extreme right is the outlet port and was attached to a regulated vacuum line via a micropipet. Figure 3C was obtained soon after the vacuum was turned on and shows that all three vertical holes in the PDMS cover get filled up immediately with the dye solution, which then flows into the diamond-shaped inlet ports underneath. The dye solution then flows into the shorter side channels giving the periodic GLAD film microstructure a darker appearance due to wetting. Figure 3D was obtained when the liquid flow front was around the midpoint of the main channel. The flow rate of the dye solution was ~2 cm/s. No evidence of leakage into the aperiodic GLAD film background is observed after emptying the inlet ports for 8 min. Similar flow experiments using fluorescent microspheres ~50 nm in diameter revealed no evidence of leakage into the aperiodic film background. The critical pressure drop that the PDMS cover could handle was measured to be in the range of ~15−20 psi. The microcolumns inside the channel were found to have similar mechanical properties as regular periodic GLAD films with a measured stiffness of ~20 $N \cdot M^{-1}/\mu M^2$.[11] The stiffness of the aperiodic background was 1 order of magnitude higher. Overall, the microstructures inside the channels were found to be stable under the conditions of liquid flow experiments.

In summary, microfluidic structures were fabricated by a new GLAD method with thin film porosity engineering capabilities. The new approach consists only of patterning the substrate with an array of protrusions followed by a single bottom-up thin film deposition step. Fluidic structures in a wide variety of materials may be fabricated using this technique, which greatly expands the options for molecule/material interactions and biocompatibility as compared with the limited set of materials employed in top-down processing in traditional fluidic architectures. Observation of pressure-driven liquid flow through the high-density microcolumn arrays indicate the feasibility of microfluidic analysis using devices made with the new GLAD method.

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