

## Supporting Information

### Abrupt Interfacial Transitions of Hydrophobic Polysilanes As Probed via Liquid Crystal-Assisted Stepwise Deposition

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#### 1) Brewster angle microscopy (BAM)

In-situ BAM observations were performed in the procedures described in the following paper; Seki T.; Sekizawa, H.; Morino, S.; Ichimura, K. *J. Phys. Chem. B.* **1998**, *102*, 5313. The rheological nature in the monolayered state could be visualized by BAM. The monolayers of PDHS and PMOS showed fluid and solid characters, respectively, which were just the opposite characters of the bulk samples (**Supporting Figure 1**). The BAM images were taken on an EMM633 system (Nippon Laser Electric Co.) on pure water at 20 °C under zero pressure conditions. The dark areas correspond to bare water surfaces.

#### 2) Atomic force microscopy (AFM)

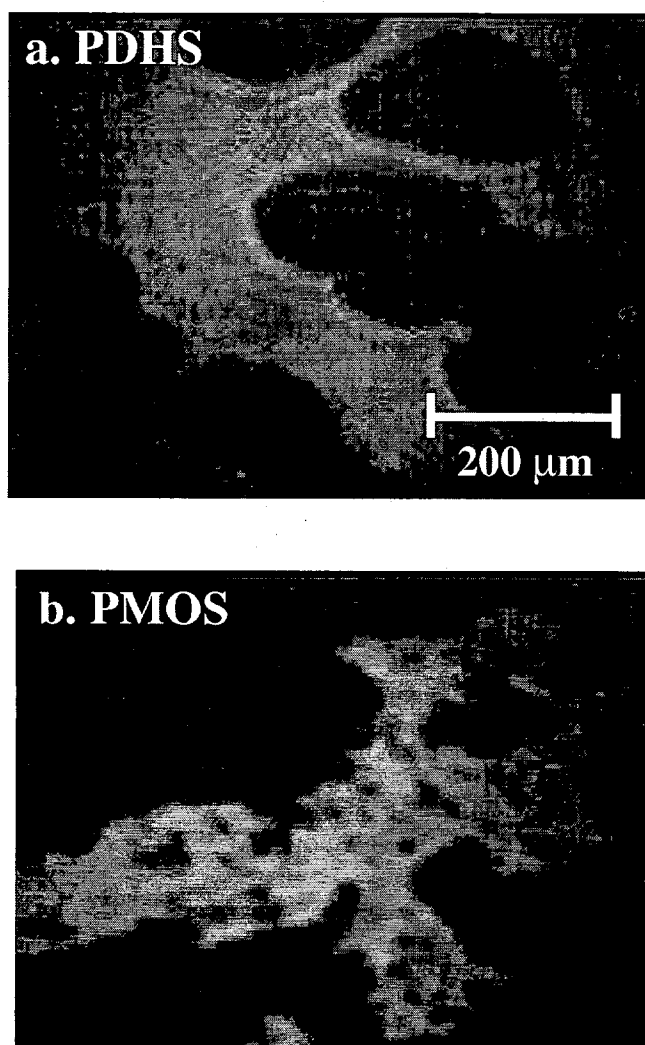
Topographical atomic force microscopic image of PDHS/5CB monolayer ( $R = 1.5$ ) transferred on a freshly cleaved mica at  $8 \text{ mN m}^{-1}$  is shown in **Supporting Figure 2**. The image was taken in the non-contact mode.

#### 3) Some remarks on the film preparation process.

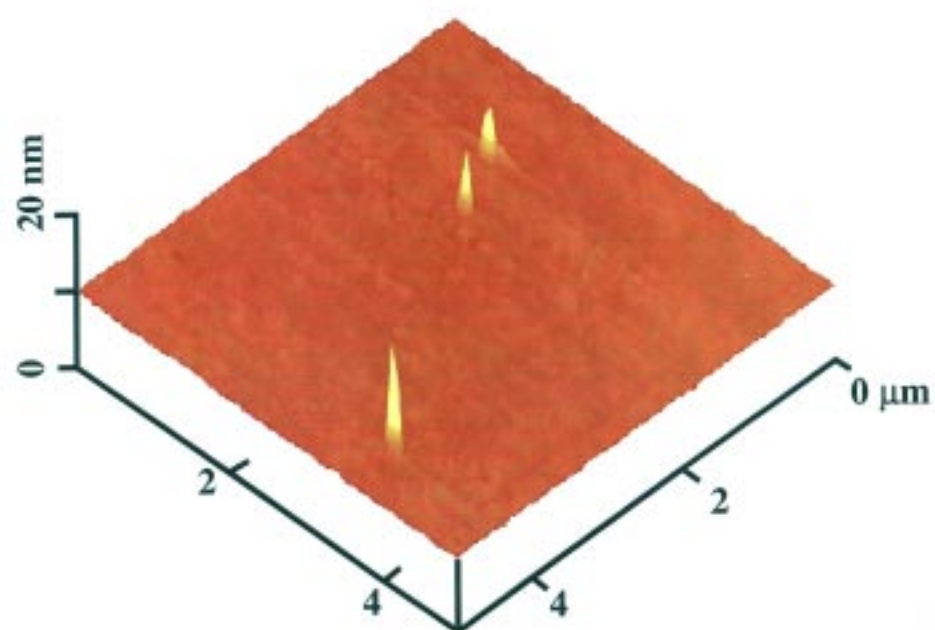
The transfer of a single layer could be achieved onto both hydrophilic

(mica or clean quartz plate, the static contact angle of water,  $\theta < 20^\circ$ ) and hydrophobic (quartz plate treated with hexamethyldisilazane,  $\theta = 81 \pm 1^\circ$ ) surfaces, however multilayer deposition was successful only for the hydrophobic surface.

The exclusion of 5CB from the deposited LB films on the hydrophobic surface was confirmed spectroscopically. The absorption band at 280 nm of 5CB was completely absent in both deposited LB films and a washing hexane solution of the film. The latter fact rules out the possibility of orientational changes of 5CB in the film. AFM images of multilayers (3 – 6 layers) of PHHS were taken on a quartz plate. Quartz plates were used here to confirm regular enhancements of absorbance with the deposition numbers. The quartz surface had ca. 10 nm surface undulations, and the roughness of the multilayers did not exceed the magnitude of substrate undulations. These facts properly support the successful achievement of layer-by-layer deposition as expected.



**Supporting Figure 1** Nagano and Seki



**Supporting Figure 2** Nagano and Seki.