

Research Article

Ultrahydrophobicity of Polydimethylsiloxanes-Based Multilayered Thin Films

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The formation of polydimethylsiloxanes (PDMSs)-based layer-by-layer multilayer ultrathin films on charged surfaces prepared from water and phosphate buffer solutions has been investigated. The multilayer films prepared under these conditions showed different surface roughness. Nanoscale islands and network structures were observed homogeneously on the multilayer film prepared from pure water solutions, which is attributing to the ultrahydrophobic property of the multilayer film. The formation of nanoscale islands and network structures was due to the aggregation of PDMS-based polyelectrolytes in water. This work provides a facile approach for generating ultrahydrophobic thin films on any charged surfaces by PDMS polyelectrolytes.

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1. Introduction

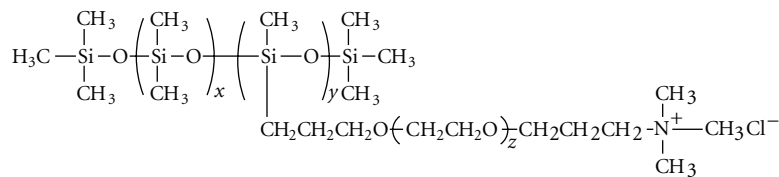
Ultrahydrophobic surfaces are now in demand of many industrial processes and of interests to biological researches [1, 2]. The ultrahydrophobic surface allows the drops of water to bead up on the surface and freely roll off the surface. Water contact angle measurement is commonly used to characterize surface hydrophobicity. Water contact angles greater than 150° have been achieved on different ultrahydrophobic surfaces [3–6].

In general, the two requirements [3] for ultrahydrophobic surfaces are the hydrophobic characteristics of the materials and the presence of highly porous network textures or nano- to microscale projection areas on the surfaces. The hydrophobic nano/microscale projections create a rough surface texture that sufficiently lowers the surface tension to reach the ultrahydrophobicity. Our results in this work were developed based on this hypothesis. However, it should be noted that hydrophilic materials could also form ultrahydrophobic surface [7, 8] and the causes of ultrahydrophobic phenomenon are still under extensive investigation.

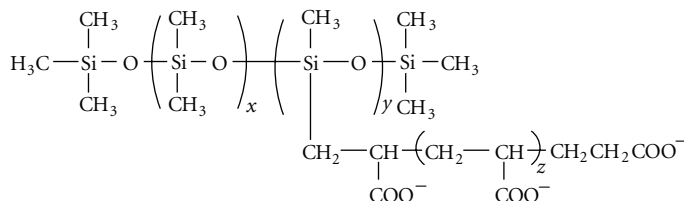
The microscale projections have been delicately micro-machined [4–6] using photolithographic methods or deposition [9–13], and the highly porous textures [3, 14–19] could

be introduced by evaporation, acidification, photochemistry process, and so forth. Most of the hydrophobic materials used for developing ultrahydrophobic surfaces were per-fluorocarbons because of their highly hydrophobic nature. Several other hydrophobic materials for creating ultrahydrophobic surfaces include polypropylene, [3] polyethylene, [20], alkylketene dimmer [21], and so forth.

Polydimethylsiloxanes (PDMSs) are industrially important materials that have been used for adhesives, sealants, lubricants, as well as in defoaming agents, damping fluids, heat transfer fluids, cosmetics, and other applications [22]. Besides its characteristically viscoelastic property, PDMS is also known as a hydrophobic material. Ultrahydrophobicity has been achieved on a microfabricated PDMS surfaces featured with 5-micron-wide pillars [23]. In this paper, we report a facile approach to generate an ultrahydrophobic surface by layer-by-layer (LbL) self-assembling a PDMS-based multilayer thin film on virtually any surfaces. LbL method [24] allows formation of ultrathin organized films on almost any metal or nonmetal surfaces through alternate adsorption of oppositely charged components, such as linear polycations and polyanions. The oppositely charged species are held together by strong ionic bonds and form long-lasting, uniform, and stable films that are often impervious



SCHEME 1: PDMS-1.



SCHEME 2: PDMS-2.

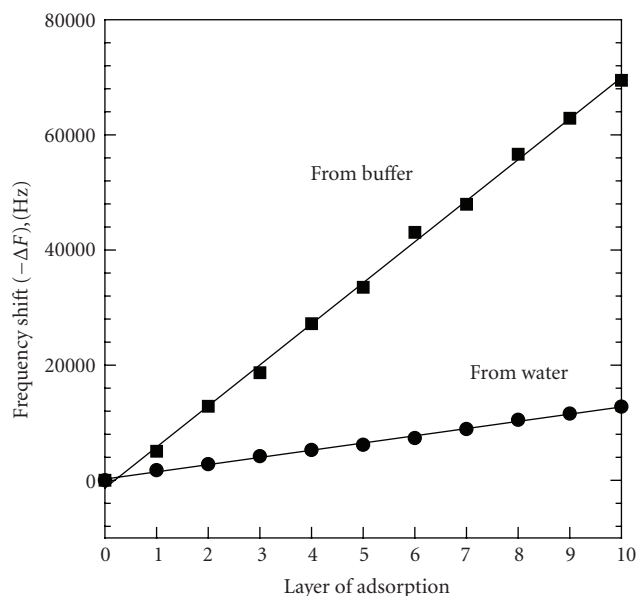


FIGURE 1: QCM resonator frequency shifts upon deposition of each layer of PDMS-1 (odd numbers) and PDMS-2 (even numbers) prepared from water and buffer solutions.

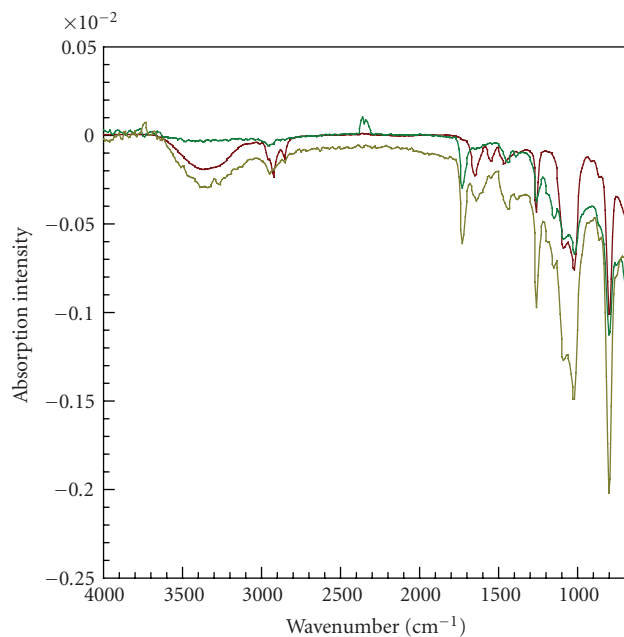


FIGURE 3: ATR spectra for PDMS-1 deposition, PDMS-2 deposition, and the 12 PDMS-1/PDMS-2 bilayer film on gold surfaces.

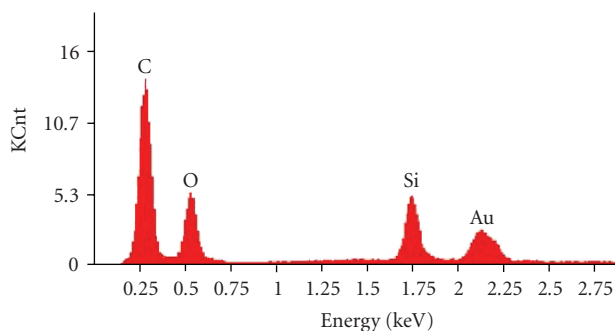


FIGURE 2: Electron dispersive spectrum (EDS) of a 12 PDMS-1/PDMS-2 bilayered thin film on a gold surface.

to a solvent. Recently, LbL self-assembly technique has been applied to develop ultrahydrophobic films [25–28]. In these ultrahydrophobic films, pre- or posttreatments were conducted to introduce micro- and nanoscale textures. In this paper, the PDMS thin film was prepared by using a simple LbL process based on PDMS polyelectrolytes without pre- or posttreatments. The surface characteristics of these PDMS-based multilayer thin films prepared from pure water and buffer solutions were investigated.

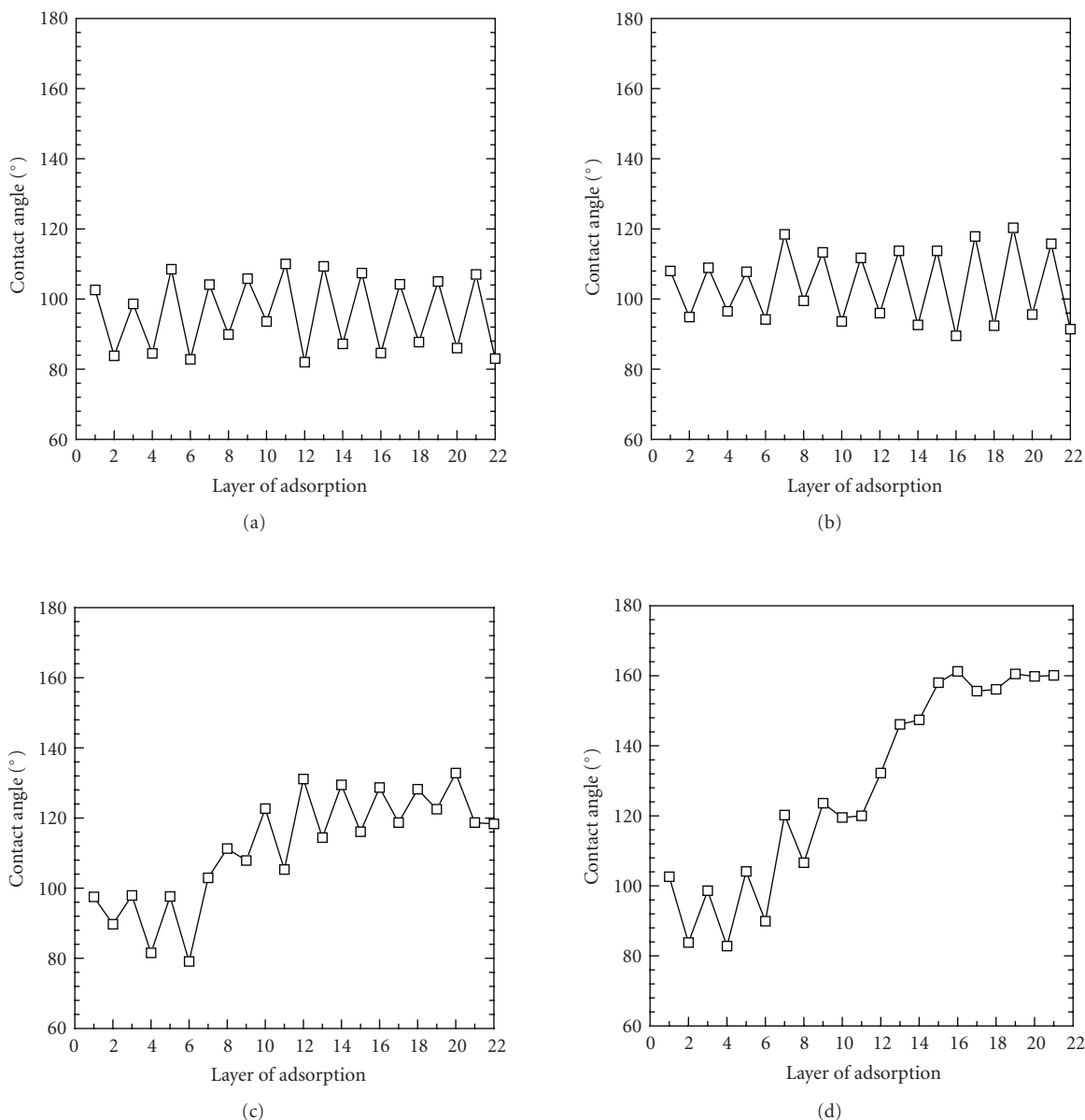


FIGURE 4: Static contact angles of water droplets on the alternately deposited PDMS-1/PDMS-2 (old numbers for PDMS-2 and even numbers for PDMS-1) multilayer film after each layer of deposition prepared from 0.025 M phosphate buffer (a), 0.005 M phosphate buffer (b), 0.001 M phosphate buffer (c), and water solution (d).

2. Experimental

2.1. Materials. Poly[dimethylsiloxane-co-methyl(3-hydroxypropyl)siloxane] graft-poly(ethyleneglycol) [(3-trimethylammonia)propylchloride]ether (PDMS-1, MW~4000), and poly(dimethylsiloxane)-graft-polyacrylates (PDMS-2, MW 26,000), poly(ethylenimine) (PEI, MW 70,000), sodium poly(styrenesulfonate) (PSS, MW 70,000), 11-mercaptoundecanoic acid, and aminopropyl triethoxysilane (ATS) were used as obtained from (Sigma-Aldrich Co. Milwaukee, USA). The structures of the PDMS-containing polycationic PDMS-1 and polyanionic PDMS-2 are shown in Schemes 1 and 2.

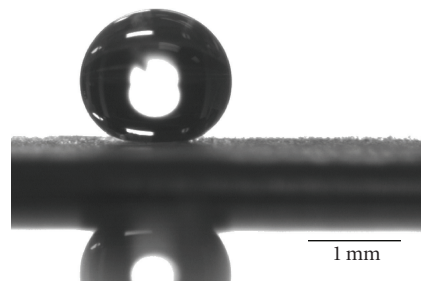


FIGURE 5: Image of a water droplet on a 12 PDMS-1/PDMS-2 bilayered thin film prepared from water solutions. The contact angle is 159.4°.

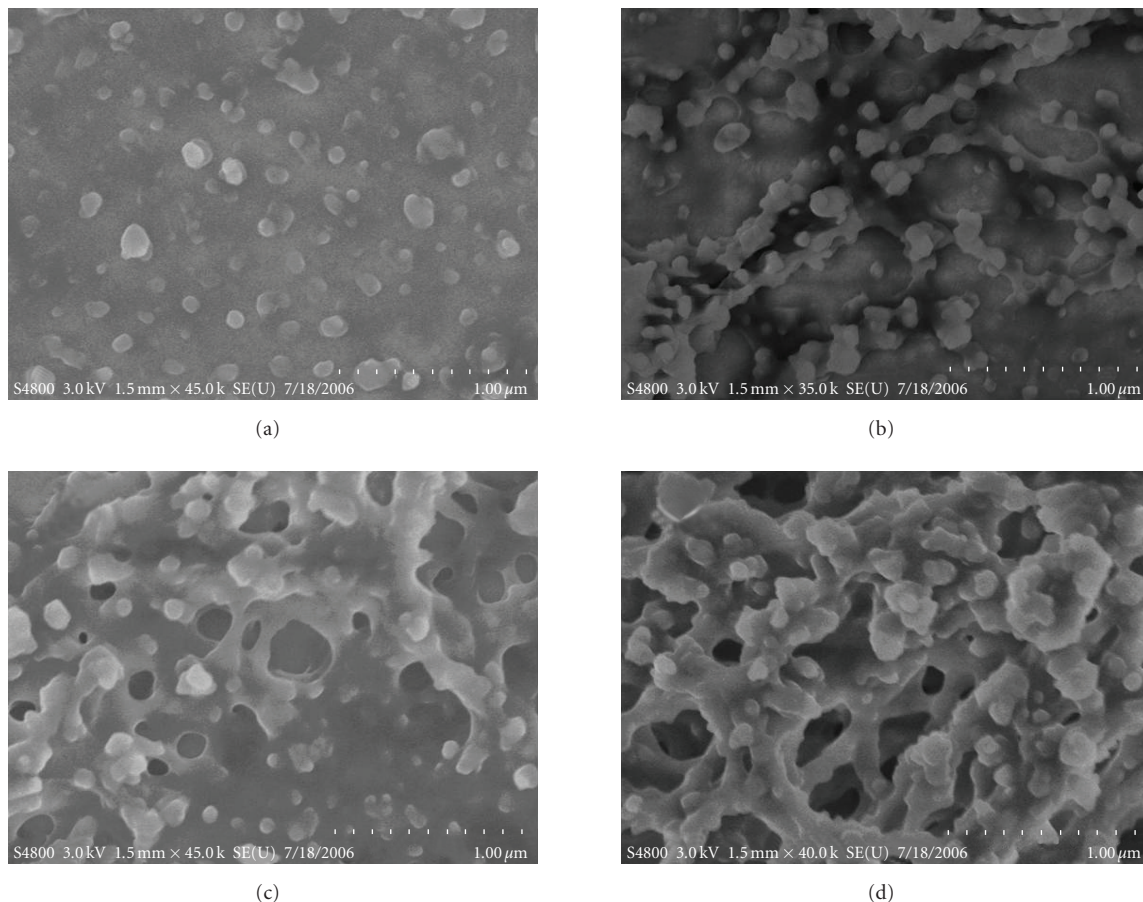


FIGURE 6: Scanning Electron Micrographs of (a) 2, (b) 6, (c) 12, and (d) 16 PDMS-1/PDMS-2 bilayered film prepared from water solutions.

2.2. Instrument. The LbL assembly process was measured by quartz crystal microbalance technique (QCM, Agilent 53131A 225 MHz) and water contact angle instrument. In a QCM measurement, the resonator was immersed in a polyelectrolyte solution for 20 minutes and dried, and the frequency change was measured in air. The long-term stability (several hours) of the quartz resonator frequency was within ± 2 Hz. All experiments were carried out in an air-conditioned room at a temperature of ca. 20°C . The resonators were covered by evaporated silver electrodes (0.16 cm^2) on both surfaces, and their resonance frequency was 9 MHz (AT-cut). The contact angle measurements were taken by an OCA15 contact angle meter (Future Digital Scientific, Long Island, USA). The OCA software provided a preset ellipse fitting for accurate contact angle calculation after the image was recorded. Atomic force microscope measurements were conducted using a tapping mode of a Quesant AFM (Model-Q-Scope250). Scanning electron micrograph (SEM) and energy dispersive spectroscopy (EDS) measurements were conducted on a Hitachi S4800 SEM. The dynamic light scattering (DLS) results were measured with an automated light scattering system (Precision Detectors ALS4000, Bellingham, Mass, USA). Attenuated infrared (ATR) spectra were recorded by using a Varian Nexus 470 ATR spectrometer.

2.3. LbL Process. The LbL procedure used for surface modification in these experiments was as follows. (A) The concentrations of the polyelectrolytes used in these experiments were 3 mg/mL PEI, 3 mg/mL PSS, 1 mg/mL PDMS-1, and 1 mg/mL PDMS-2. Polyelectrolytes, except for PDMS-2 solutions were directly prepared in pure water or various phosphates buffer solutions (pH 7.0). The PDMS-2 was firstly dissolved in acetone at 10 mg/mL as a stock solution and then diluted 10 folds in water or buffer solutions. (B) A substrate carrying a surface charge was alternately immersed in the oppositely charged polyelectrolyte solutions. Each immersion took 20 minutes with 1 minute intermediate water washing. (C) This cycle was repeated several times until the desired number of multilayer films was reached. The assembled multilayer films prepared both from water and buffer solutions were investigated to compare the uniformity and surface characteristics.

3. Results and Discussions

Silver electrodes have a naturally negative charge on their surfaces. 11-Mercapto-undecanoic acid and ATS were used to develop a charged monolayer film on the gold and silicon (or glass) surfaces, respectively, using typical surface modification procedures [29, 30]. These monolayer films

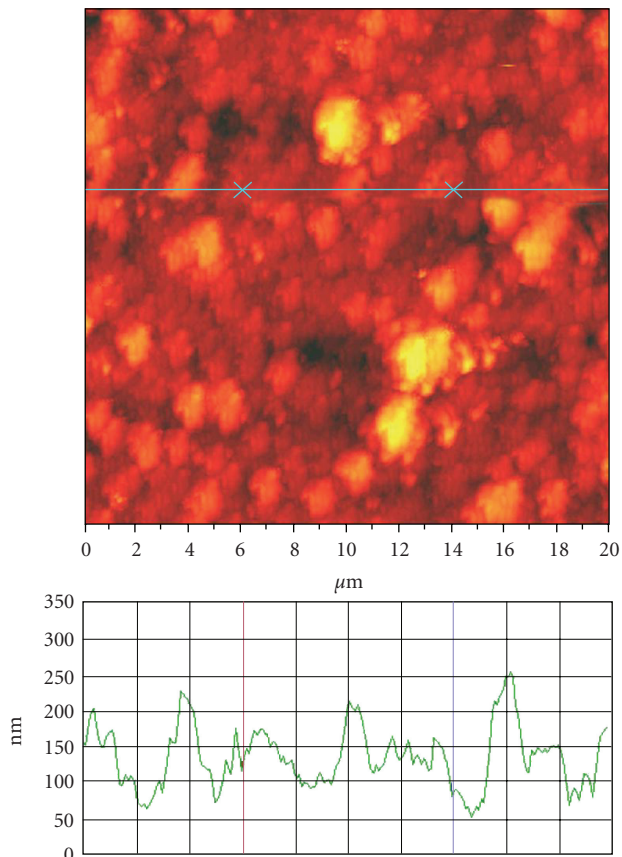


FIGURE 7: An AFM image of a 16 PDMS-1/PDMS-2 bilayered film prepared from water solutions. Z scale was in nm.

introduced a layer of charges on the gold or silicon surfaces that could strengthen the stability of the consequent LbL assembly.

In the LbL assembly process, three bilayers of PEI/PSS were firstly formed on the surfaces that provided a solid base for further immobilization. Multiple bilayers of PDMS-1/PDMS-2 were formed on the top of these layers.

The results showed that the PDMS-1/PDMS-2 multilayer films on Ag, charged Au and SiO₂ surfaces had virtually the same properties when they were prepared under the same conditions. However, the films prepared from water solutions showed significantly different characteristics from those prepared from buffer solutions, especially those from higher concentration of buffer solutions.

The QCM experiments showed the formation of the PDMS-1/PDMS-2 multilayer film on the silver-covered quartz crystal resonators (Figure 1). The frequency decreases of the resonators as a function of multilayer numbers were nearly linear for the multilayer films prepared from both water solution and a 0.025 M phosphate buffer solution. The frequency change, however, varied under these two conditions. The deposition of each polyion bilayer in water and in the 0.025 M buffer solutions resulted in approximately 1.6 kHz and 7 kHz frequency decrease (ΔF), respectively. These were corresponding to 1.44×10^{-6} g and 6.30×10^{-6} g

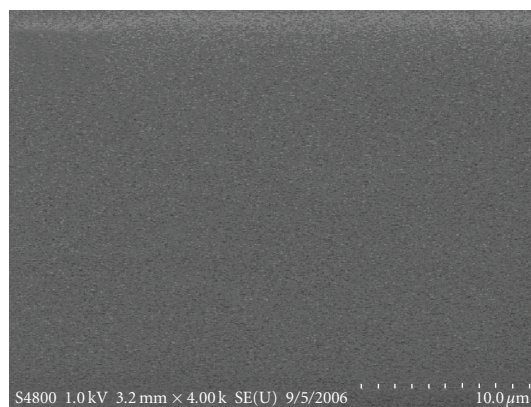
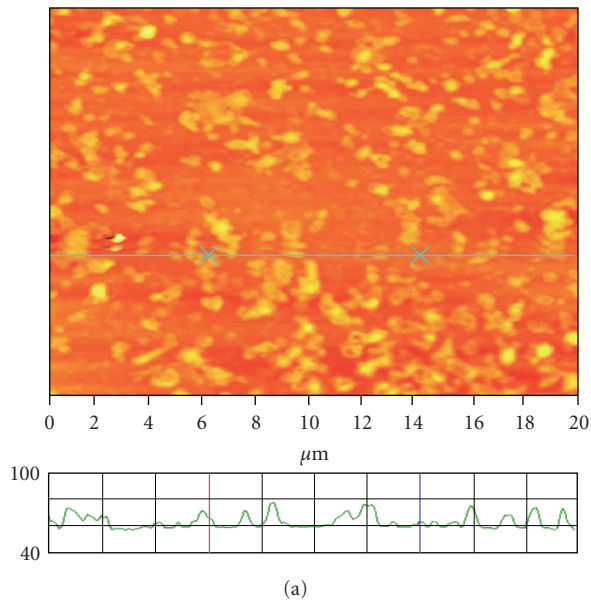


FIGURE 8: (a) AFM and (b) SEM images of a 12 PDMS-1/PDMS-2 bilayered film prepared from a 0.025 M buffer solutions. Z scale was in nm.

of mass increase on the resonator surfaces for the films from water and from buffer solutions, respectively, after each polyion bilayer deposition according to the following equation [31]:

$$\Delta F = -1.832 \times 10^8 M/A, \quad (1)$$

where A is the surface area of the resonator (in the unit of cm²), M is the adsorbed mass, and ΔF is the frequency shift of the quartz resonator.

The EDS result (Figure 2) of a 12 PDMS-1/PDMS-2 bilayer film on a gold surface verified the formation of the PDMS film. The film contains 13.2% of Si, 61.3% of C, and 13.2% of O atoms.

Figure 3 shows ATR spectra for PDMS-1 deposition, PDMS-2 deposition, and the 12 PDMS-1/PDMS-2 bilayer film on gold surfaces. All these figures showed four characteristic PDMS peaks: the bands at 1260 cm⁻¹ (Si-CH₃ symmetric bend), 1100 cm⁻¹ (Si-O-Si symmetric stretch),

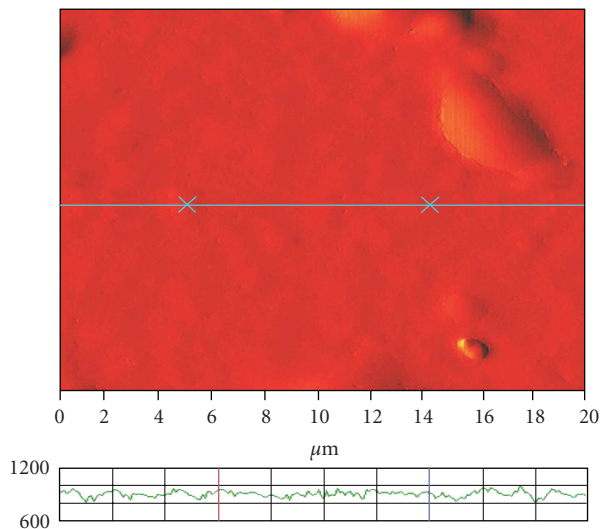


FIGURE 9: An AFM image of a 16 PDMS-1/PDMS-2 bilayered film prepared from water solutions after a 2-hour heating at 180°C .

1060 cm^{-1} (Si–O–Si asymmetric stretch), and 800 cm^{-1} (CH_3 rocking) [32]. PDMS-2 also showed a characteristic sharp peak for carbonyl group at 1740 cm^{-1} . The ATR spectrum of the multilayer film was an addition of spectra of PDMS-1 and PDMS-2, confirming the formation of PDMS-1/PDMS-2 bilayer composites.

The static water contact angle experiments not only confirmed the formation of the PDMS multilayer films, but also demonstrated the ultrahydrophobicity of multilayer surfaces prepared under controlled conditions. For the multilayer films prepared from 0.025 M buffer solutions, the contact angle change showed a regular pattern (Figure 4(a)). The contact angles, obtained when the PDMS-2 was at the outmost layer ($105 \pm 5^{\circ}$), were approximately 20° larger than those obtained when PDMS-1 was at the outmost layer ($85 \pm 5^{\circ}$). The 20° contact angle difference may attribute to the polarity difference between PDMS-1 and PDMS-2. There were approximately 80 wt.% of the polar polyethylene glycol component in PDMS-1, while only 20 wt.% of polar polyacrylates component in PDMS-2. The contact angles of water droplets on a PDMS multilayer film prepared from 0.005 M buffer solutions (Figure 4(b)) were similar to those obtained from the 0.025 M buffer solutions. The contact angles of water droplets on a PDMS multilayer film prepared from 0.001 M buffer solutions showed an increase after 10 layers (Figure 4(c)). For the multilayer films prepared from the water solutions, the contact angles of water droplets on the first three-bilayered films (Figure 4(d)) showed similar pattern as those in Figure 4(a). After three bilayers, the contact angle gradually increased. The static contact angles reached a plateau at approximately 160° after 8 bilayers as shown in Figure 4(d), and the water droplets bead up on the surface and could freely roll off the surface without becoming pinned (Figure 5). The contact angles remained at 160° after 8 bilayers when either the PDMS-1 or the PDMS-2 was at the outmost layer. The ultrahydrophobic surface is very

stable and the water droplet can sit on the surface for hours (before all evaporated) with low contact angle hysteresis [33], indicating ultrahydrophobic characteristics of this PDMS thin film. The ultrahydrophobic surface is stable and resistant to strong acids, bases, and organic solvents. The contact angles were the same at approximately 160° after one-hour immersing either in a 1 N HCl or a 1 N NaOH. Similar results were observed after exposing the film to organic solvents, including ether, THF, acetone, CH_2Cl_2 , benzene, and hexane. However, the film can be readily damaged by mechanical forces, such as scratches.

These contact angle differences and the ultrahydrophobic phenomenon could be explained by the surface roughness as shown from AFM and SEM images in Figures 6, 7, and 8. For the multilayer film prepared from water solutions, SEM images showed the appearance of nanoparticles on the surfaces after two PDMS-1/PDMS-2 bilayers (Figure 6(a)); these particles accumulated and connected after six PDMS-1/PDMS-2 bilayers (Figure 6(b)). Islands and network structures at nanometer-scale were formed after 12 PDMS-1/PDMS-2 bilayers (Figures 6(c)-6(d)). The depth of the cavities on the 16 PDMS-1/PDMS-2 bilayered films ranged from 200 to 500 nm (Figure 7). The nanometer-scale islands, with diameters at approximately 100 nm, were widely distributed on the surfaces. In comparison, a 12 PDMS-1/PDMS-2 bilayered film prepared from the 0.025 M buffer solutions was rather smooth with a 20 nm roughness (Figure 8). The root-mean-square average surface roughness data from each AFM measurement of 16 PDMS-1/PDMS-2 films prepared from 0.025, 0.005, 0.001 M PBS, and water were 20 ± 2 , 24 ± 2 , 106 ± 8 , and 180 ± 25 nm, respectively.

After rinsing the multilayer film samples from water, no salts were observed on the surfaces using EDS measurements. The formation of nanometer-scale islands and network structures on the surfaces could be explained by the aggregation of PDMS-1 and PDMS-2 in pure water measured from dynamic light scattering experiments. The average particle sizes of PDMS-1 and PDMS-2 in pure water were 156 ± 50 nm and 60 ± 40 nm in diameter, respectively, which could also be supported from the SEM images as shown in Figure 6(d). By comparison, in the 0.025 M buffer solutions, the average particle sizes of PDMS-1 and PDMS-2 were 40 ± 20 nm and 15 ± 10 nm, in diameter, respectively, suggesting much less aggregation than those in buffer solutions, which may be due to less salvation energy of polyelectrolytes in high ionic strength solutions [34].

The ultrahydrophobic film is resistant to acids and bases and the ultrahydrophobicity remains after half a year in ambient conditions. However, heat will convert the film to a uniform and smooth surface (Figure 9), which loses its ultrahydrophobic characteristics. The contact angle of this film after heat was $102 \pm 4^{\circ}$.

4. Conclusions

In summary, this work demonstrates the formation of polydimethylsiloxanes (PDMSs) based layer-by-layer multilayer thin films on charged surfaces. The multilayer films prepared

from water and phosphate buffer solutions showed different surface characteristics. Ultrahydrophobic phenomenon was observed on the multilayered thin films prepared from water solutions due to the formation of nanostructured network surface. Dynamic light scattering experiments showed that both PDMS-1 and PDMS-2 aggregated in pure water, which could be attributed to the formation of nanometer-scale islands and network structures on the surfaces. This work provides a facile approach to develop an ultrahydrophobic film on possibly any charged surfaces without pre- or posttreatments. The ultrahydrophobic surface is stable and resistant to acids and bases, but will be readily damaged by heat and mechanical forces.

Acknowledgments

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