

SiO_x Deposition on Polypropylene-Coated Paper With a Dielectric Barrier Discharge at Atmospheric Pressure

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Abstract—Deposition of transparent oxide films at atmosphere pressure onto polymeric substrates at room temperature is a technique gaining rapid acceptance. These films can be used for numerous applications such as antiscratch and water permeation barrier coatings. In this paper, a $\sim 1.4\text{-}\mu\text{m}$ SiO_x layer has been deposited on polypropylene-coated paper at atmosphere pressure with a filamentary dielectric barrier discharge (DBD). The DBD linear discharge was driven by a 30-kHz power supply. Scanning electron microscope, X-ray photoelectron spectroscopy, and attenuated total reflection-Fourier transform infrared spectroscopy were used to characterize the deposited film. The deposited thin film decreased water permeation through the paper by $\sim 15\%$.

Index Terms—Atmospheric pressure plasma deposition, dielectric barrier discharge (DBD), polypropylene (PP), SiO_x film.

I. INTRODUCTION

POLYMERS have been widely used in industry for its flexibility, printability, and recyclability [1]. However, some properties of polymers such as poor gas barrier and antiscratching performances may limit their applications. As a result, SiO_x films deposited on polymer substrates are drawing extensive attention, because they can act as transparent and microwaveable gas/moisture barrier layers and scratch-resistant coatings [2]–[4].

The polymeric substrate cannot withstand high temperature, hence requires the SiO_x film to be deposited at

relatively low temperatures. Plasma-enhanced chemical vapor deposition is a promising method [1], [5]–[7] because of the high deposition rate and the low substrate temperature during the process. However, these processes are generally carried out in low-pressure systems, which require bulky and expensive equipment to sustain the vacuum. This can dramatically increase the cost of final product even though there are no technical limitations for the method to be used in industry. The recent advance in atmospheric pressure plasma deposition technology [8], [9], especially utilizing dielectric barrier discharge (DBD) [10]–[14], unlocked a new way for the SiO_x coating. Different discharge regimes of DBD including Townsend discharge [10], [11], filamentary discharge [12], and glow discharge [13], [14] were used to deposit SiO_x films onto polymer substrates. It is undeniable that both the Townsend and the glow discharge modes ($10^3\text{--}10^4\text{ W/m}^2$) cannot deposit as much power density into the plasma as the filamentary mode ($>10^5\text{ W/m}^2$). Besides, they are very sensitive to small changes in the operating conditions and easy to transfer toward the filamentary discharge [15]. Hence, filamentary DBD was adopted in this paper.

Many research groups reported successful experimental results for deposition of SiO_x at atmospheric pressure on 10–100- μm thick and polymeric foils [2], [16]–[19]. The deposition rates and film properties are greatly affected by the substrate temperature [11] and the processing parameters such as the scanning speed of the torch, the distance from the torch to the substrate [4], the type of precursors [18], and the flow rates of the precursor [12]. Nevertheless, SiO_x deposited on polyethylene terephthalate, polyethylene-2, 6-naphthalate, and polycarbonate are more intensely investigated in the literature, and polypropylene (PP) seems to be more problematic than polyesters as base material [6], [7]. The challenges of deposition technique on PP not only lie in the low temperature resistance but also in the relatively high thermal expansion coefficient of PP substrate which can cause cracks on the SiO_x coatings [4] if the process temperature is too high.

In this paper, we intended to deposit a crack-less SiO_x film on a thin PP layer ($\sim 1.6\text{ }\mu\text{m}$) covered on $\sim 70\text{-}\mu\text{m}$ -thick paper using filamentary DBD at atmospheric pressure and room temperature. The transition voltage between crack-less and cracked film was reported. The current-voltage characteristic has been monitored during the deposition process to confirm the discharge regime. After deposition,

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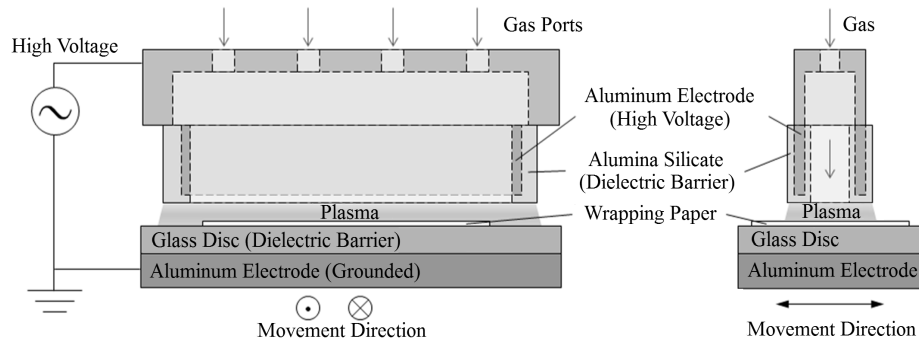


Fig. 1. Parallel-plate type DBD setup schematic. The high-voltage electrode was supported by structural framing at a fixed height, and the ground electrode was put on the worktable which moved with a speed of 1 mm/s during the process.

the film surface and cross section were observed using scanning electron microscope (SEM) to get the surface morphology and the deposition thickness. It showed that a SiO_x film without cracks was obtained. Furthermore, the chemical composition and the bonds of the deposition film were measured by X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FTIR).

II. EXPERIMENTAL SETUP

The parallel-plate type DBD plasma source is schematically presented in Fig. 1. The discharge electrodes were both made of aluminum. The top electrode was an inverted U-shape high-voltage electrode, designed to also supply processing gas into the 2-mm discharge gap. It was covered with a 2-mm-thick dielectric barrier made of alumina silicate, and was connected to a power supply capable of providing ac power signals with frequencies from 25 to 35 kHz and voltages from 5 to 15 kV. We took the same DBD power supply used in [20] and adapted it to this new geometry. The bottom electrode was grounded and covered with a 5-mm-thick glass disk as dielectric barrier. The electrode configuration provided an approximate discharge area of $140 \text{ mm} \times 35 \text{ mm}$. The ground electrode was attached to a one-axis motor-driven worktable to process large samples. The worktable could move up to 250 mm with a maximum speed of 15 mm/s. The whole system was operated at atmospheric pressure. Neither the ground electrode nor the high-voltage electrode had any additional cooling.

The SiO_x film was deposited on a paper sheet which was precovered with a $\sim 1.6\text{-}\mu\text{m}$ PP layer. The $140 \text{ mm} \times 80 \text{ mm}$ paper was attached to the glass disk using Kapton tape at the center region of the ground electrode. Hexamethyldisiloxane (HMDSO) was used as the precursor during the deposition process. HMDSO is a common monomer in the deposition of SiO_x [2], and the process of HMDSO decomposition was well-described elsewhere [21], [22]. A venturi pump was used to turn the liquid HMDSO into vapor, and a needle valve was used to control the flow rate of HMDSO. The He/air/HMDSO gas mixture was provided to the discharge area through the four gas ports on the high-voltage electrode. Helium was supplied to obtain stable discharge, and compressed air was utilized to get more inorganic characteristics on the SiO_x film by promoting carbon oxidation of HMDSO [23].

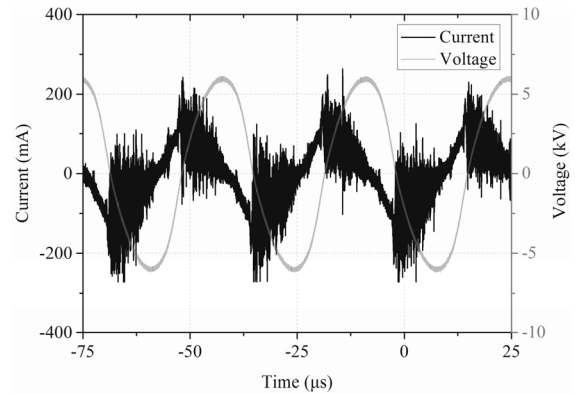


Fig. 2. Voltage–current curve for the deposition process. It indicated a classical filamentary DBD.

Thin SiO_x films were deposited on the PP-coated paper in the 30 kHz discharge in two phases. During the first phase—pretreatment phase, pure helium (10 L/min) plasma was run for 10 min to improve adhesion. During the second phase—deposition phase, 1 L/min of compressed air and 0.045 mL/min of HMDSO were added to the helium discharge for 20 min. To compensate for the inevitable inhomogeneous characteristics of filamentary discharge, the worktable was moving back and forth with a speed of 1 mm/s during the whole process. The SiO_x film could be got within the length of high-voltage electrode and the scanning distance of ground electrode. It was expected that the size of the plasma source could be scaled up for larger substrates in industry. In addition, the experiments were repeated for many times to make sure that the deposition results were not occasional but reproducible.

III. RESULTS AND DISCUSSION

A. Dielectric Barrier Discharge Regime

During the deposition process, the voltage–current discharge characteristics were measured with high-voltage probe (Tektronix P6015A 1000X) and Pearson current monitor (Model 110), and stored on oscilloscope for postprocessing. In the experiments, 7 kV was a common applied voltage, and the voltage–current curve was shown in Fig. 2. A large number of short-lived microdischarges were observed on the

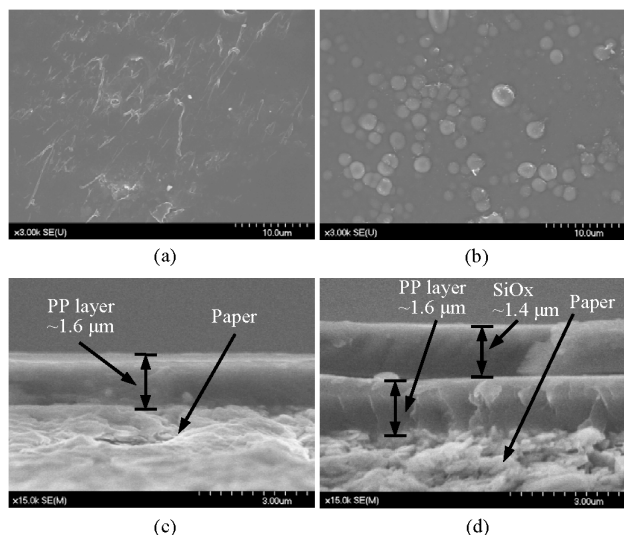


Fig. 3. SEM images of SiO_x layer on PP-coated paper. (a) Surface before deposition—typical PP morphology. (b) Surface after deposition—microparticles on SiO_x film. (c) Cross section before deposition—a $\sim 1.6\text{-}\mu\text{m}$ -thick PP layer on the paper. (d) Cross section after deposition—a $\sim 1.4\text{-}\mu\text{m}$ -thick SiO_x layer on the PP layer.

current curve per half-cycle of the applied voltage, which indicated a classic filamentary DBD. It has already been proved that continuous and uniform films can be formed by atmospheric pressure filamentary plasma [12], [24]. In this paper, the localized thickness uniformity of the SiO_x film could be observed using SEM.

B. Surface and Cross-Sectional Morphologies

The SiO_x coating thickness on polymer was generally obtained indirectly by means of stylus profiler or ellipsometry measurement of film deposited on silicon wafer [4], [11], [22]. However, the absolute film thickness on polymers cannot be obtained in this way due to its softness. In this paper, the measurement was performed by SEM (Hitachi S4700 SEM), through observing the cross section of the PP-covered paper substrate. Since the deposited film could be easily damaged by mechanical cutting, the paper was thus dipped first into liquid nitrogen to harden it so that it could be cleaved off to make a clean cross section. SEM pictures were shown in Fig. 3 with surface and cross-sectional images of the substrate before and after deposition when the applied voltage was 7 kV. Before each measurement, the substrate was bended into a 5-mm-radius curve to test the crack resistance.

From Fig. 3(a) and (c), the $\sim 1.6\text{-}\mu\text{m}$ -thick PP layer on the paper could be seen before the deposition process started. In contrast, in Fig. 3(b) and (d), the $\sim 1.4\text{-}\mu\text{m}$ -thick SiO_x layer could be seen above the PP layer after deposition. From this, the deposition rate was calculated to be $(70 \pm 7) \text{ nm/min}$ and $(0.8 \pm 0.08) \text{ mm}^3/\text{min}$. Moreover, as shown in Fig. 3(b), there were no cracks after bending but microparticles formed on the surface. The microparticles were believed to be mainly related to the organic composition such as methyl groups in the SiO_x film. This could be confirmed by the XPS and FTIR results. The particles might also be caused by the

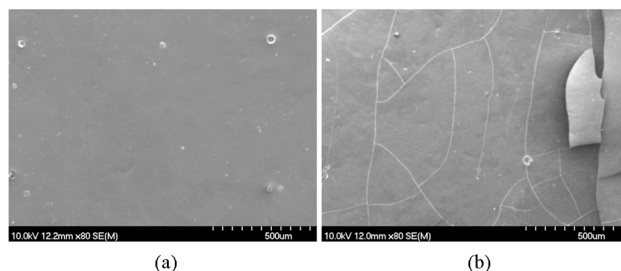


Fig. 4. SEM images of SiO_x layer on PP-coated paper. (a) Applied voltage was 7 kV. (b) Applied voltage was 8 kV.

nonhomogeneous of the filamentary discharge [25]. Also, there was another possibility that samples were affected by dust and humidity, since the experiments were not conducted in a clean room and the compressed air was directly from the university airline without any desiccation.

In addition, an experiment with applied voltage at 8 kV was carried out. As shown in Fig. 4, compared with the crack-less surface at 7 kV in Fig. 4(a), there were obvious cracks at 8 kV in Fig. 4(b). This should be mainly due to the large difference of the thermal expansion coefficients between the SiO_x film and the PP layer. When the voltage was higher, the processing temperature was higher. After the processing, when the substrate temperature returned back to room temperature, cracks appeared when 8 kV was used. Therefore, 7 kV was used most as the applied voltage in this study.

C. Chemical Composition

XPS (Kratos Axis XPS) result in Fig. 5(a) showed the elements on the substrate surfaces before and after deposition, and the energy resolution of XPS measurement was selected to be 0.5 eV/step to distinguish closely spaced peaks. The major component of the original surface was carbon ($\sim 80\%$), and there was no silicon peaks. In contrast, after deposition, silicon ($\sim 36\%$), oxygen ($\sim 45\%$), and carbon ($\sim 18\%$) peaks were observed. It was noticed that there was still $\sim 18\%$ carbon on the deposited film, which was impossible to be caused only by the surrounding dust and contamination. This meant at least one part of the carbon came from the methyl groups of HMDSO, and the SiO_x film contained a small amount of organic composition.

More specifically, narrow scans of the Si 2p, O 1s, and C 1s were analyzed. The peak shifting was calibrated by C 1s peak at 285.0 eV. As shown in Fig. 5(b), the Si 2p signal could be deconvoluted into two peaks, which were centered at 104 eV ($\sim 54\%$) and 103.2 eV ($\sim 46\%$). By comparing these individual peaks energies with the National Institute of Standards and Technology XPS database, these two peaks corresponded to the SiO₂ bonding [26] and the SiO_{1.91} bonding [27], respectively. The O 1s peak at 533.1 eV in Fig. 5(c) could be ascribed to O–H bonds (H₂O, Si–O–H) [28]. As for the C 1s narrow scan in Fig. 5(d), there was a small peak at the binding energy of 288.1 eV corresponding to the $-\text{C}=\text{O}/\text{O}-\text{C}-\text{O}$ bond [29]. All of these assignments would be further supported by the FTIR analysis discussed later.

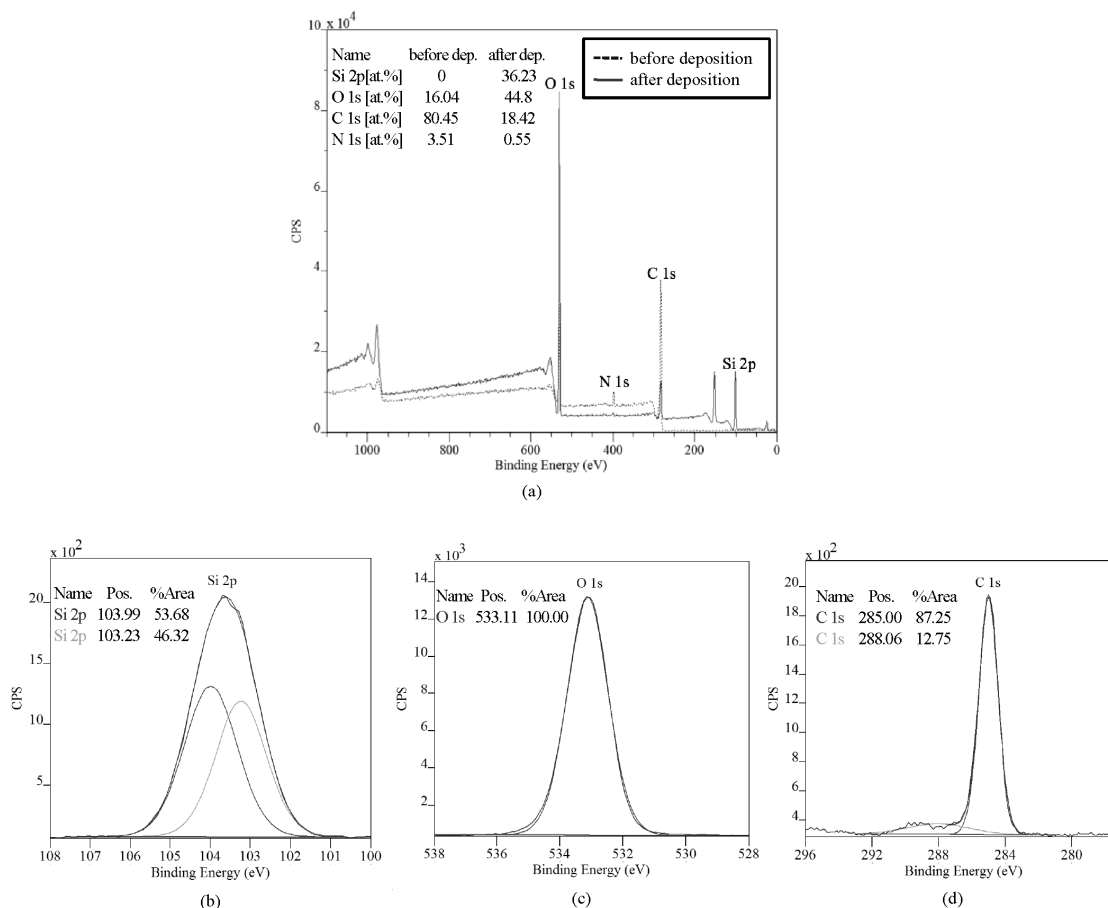


Fig. 5. XPS data. (a) Comparison of the chemical compositions between surfaces before and after deposition. (b) Narrow scan of Si 2p on the SiO_x deposition film. (c) Narrow scan of O 1s on the SiO_x deposition film. (d) Narrow scan of C 1s on the SiO_x deposition film.

The XPS result indicated that the methyl groups of HMDSO did not dissociate completely here. It has already been demonstrated that the increase of O_2/HMDSO concentration would lead to the reduction of organic groups in the films deposition process [6], [30]–[32], which could be tried in our future work.

D. Surface Chemical Bonding

To confirm the chemical bonds on the SiO_x film, the film was observed by FTIR (Thermo Nicolet Nexus 670) equipped with the ATR accessory (Nicolet smart multibounce HATR). The spectrum was obtained from an average scan of 100 scans in the range of $700\text{--}4000\text{ cm}^{-1}$ at a resolution of 4 cm^{-1} . There were no peaks beyond 1500 cm^{-1} , so only the spectrum in the range of $700\text{--}1500\text{ cm}^{-1}$ was shown in Fig. 6.

The most intensive peaks at $\sim 790\text{ cm}^{-1}$ [33] and $980\text{--}1070\text{ cm}^{-1}$ [18] could be assigned to Si–O–Si bending mode vibration. The peak in the region of $900\text{--}960\text{ cm}^{-1}$ was associated to Si–OH stretching [18]. And the Si–CH₃ peaks at 840 cm^{-1} and $1280\text{--}1260\text{ cm}^{-1}$ [corresponding to Si–(CH₃)_n groups with $n = 1, 2, 3$] [16] were clearly observed in the spectrum, which indicated the organic composition in the SiO_x film.

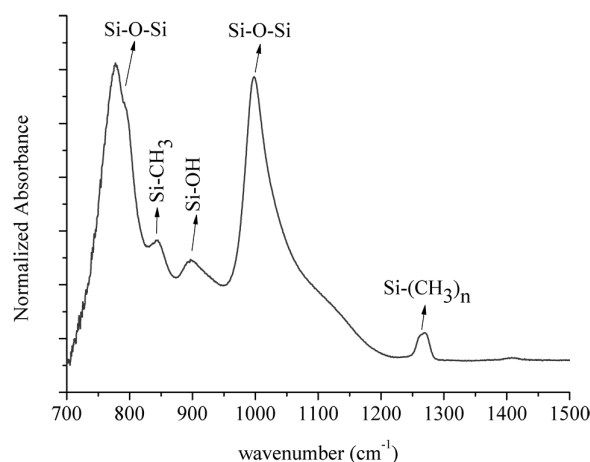


Fig. 6. FTIR data. The Si–O–Si and Si–(CH₃)_n bands existed on the SiO_x film.

E. Water Permeation Test

A homemade test device was developed to test the water permeation property of the crack-less SiO_x film, as presented in Fig. 7. The procedure can be described as follows. Water was poured into the Teflon holder into its small cylindrical tank. The holder was covered by the paper substrate, and then a plastic cover with a round hole was used to clamp the

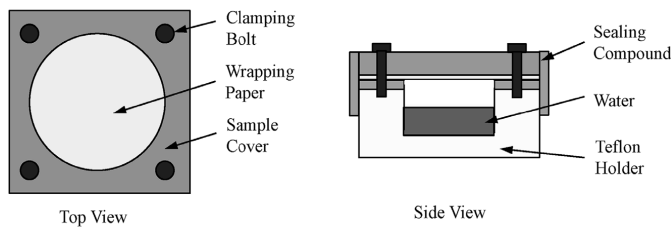


Fig. 7. Homemade water permeation test device schematic. The test setups were placed into a vacuum desiccator with Drierite as desiccant so that the water loss could be considered as the water permeation amount.

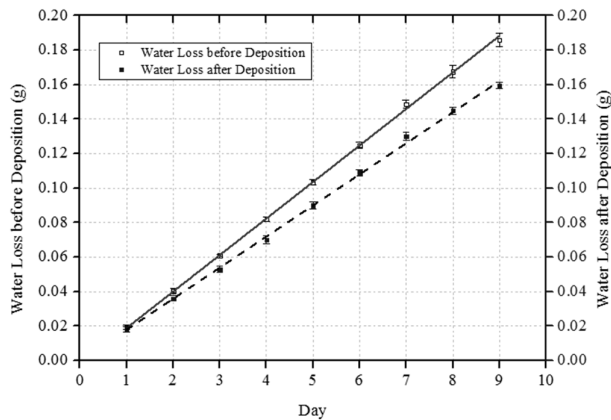


Fig. 8. Water permeation test result. The weights of the setups were weighed once a day, and the accumulated weight losses were recorded.

paper to the Teflon holder. The Teflon–paper–cover joints were sealed with sealing compound to prevent the water evaporating through the sides. The test setups were placed into a vacuum desiccator with Drierite as desiccant. To accelerate the water evaporation speed, the vacuum desiccator was put into an oven and the temperature was kept at 55 °C. The humidity in the vacuum desiccator was controlled between 15% and 18%.

Three samples before and three after deposition were used to measure water permeation over time. These Teflon holder assemblies were weighed once per day, and the mass loss during the first nine days are presented Fig. 8. The result showed that water permeation of paper with the deposited film was $(14.9 \pm 1.1)\%$ less than that before deposition. Although the resistance to water vapor improved a little bit, this was an unexpected low value for such a thick SiO_x coating on the substrate. The main problem was the water vapor pressure inside the test device pushed the substrate during the test. Since the edges were sealed tightly, the substrates could not stretch freely. Many small cracks were observed on the film after the water permeation test. It was assumed that the water resistance could be enhanced much more than 15% without the cracks. Applications which do not constrain the overall expansion of the material, such as wrapping a product, could see larger benefits.

IV. CONCLUSION

In this paper, we have presented a method for crack-less SiO_x film deposition on PP-coated paper substrate. Filamentary DBD at atmospheric pressure was used to get

low substrate temperatures, preventing degradation of the thin layer of PP. The SEM result showed that the film was locally uniform without cracks and with microparticles on surface. The film composition was confirmed by both XPS and FTIR. It was SiO_x with a small amount of organic composition caused by the methyl groups of HMDSO. Water permeation rate through substrate with the $\sim 1.4\text{-}\mu\text{m}$ SiO_x film decreased by $\sim 15\%$.

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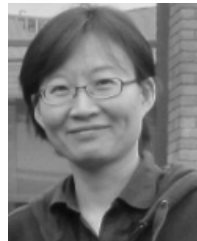
include plasma etching and deposition at atmospheric pressure.



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