

# Characteristics of $\text{SiO}_x\text{N}_y$ films deposited by inductively coupled plasma enhanced chemical vapor deposition using HMDS/ $\text{NH}_3/\text{O}_2/\text{Ar}$ for water vapor diffusion barrier

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## Abstract

$\text{SiO}_x\text{N}_y$  thin films were deposited by inductively coupled plasma enhanced chemical vapor deposition (ICP-PECVD) using hexamethyldisilazane (HMDS, 99.9%)/ $\text{NH}_3/\text{O}_2/\text{Ar}$  at a low temperature, and examined for use as a water vapor diffusion barrier. The film characteristics were investigated as a function of the  $\text{O}_2:\text{NH}_3$  ratio. An increase in the  $\text{O}_2:\text{NH}_3$  ratio decreased the level of impurities such as  $-\text{CH}_x$ ,  $\text{N}-\text{H}$  in the film through a reaction with oxygen. Thereby, a more transparent and harder film was obtained. In addition, an increase in the  $\text{O}_2:\text{NH}_3$  ratio decreased the nitrogen content in the film resulting in a more  $\text{SiO}_2$ -like  $\text{SiO}_x\text{N}_y$  film. Using  $\text{SiO}_x\text{N}_y$  fabricated with an  $\text{O}_2:\text{NH}_3$  ratio of 1:1, a multilayer thin film consisting of multiple layers of  $\text{SiO}_x\text{N}_y$ /parylene layers was formed on a polyethersulfone (PES, 200  $\mu\text{m}$ ) substrate, and its water vapor transmittance rate (WVTR) was investigated. A  $\text{WVTR} < 0.005 \text{ g}/(\text{m}^2 \text{ day})$  applicable to organic thin film transistors or organic light emitting diodes was obtained using a multilayer composed of  $\text{SiO}_x\text{N}_y$  (260 nm)/parylene ( $< 1.2 \mu\text{m}$ ) on the PES.

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*Keywords:* HMDS; Diffusion barrier; PECVD; Siliconoxynitride; Passivation; WVTR

## 1. Introduction

The permeation of water vapor and oxygen into the active layers of several display devices, e.g., organic thin film transistor-liquid crystal display (OTFT-LCD) devices, organic light emitting diodes (OLEDs), etc. can cause damage or a malfunction [1,2]. Therefore, permeation barrier films are used to enhance the lifetime of these devices. For polymer-based OLED displays, which require perfect encapsulation against the inward permeation of water and oxygen [3,4], the oxidation of the metallic cathode by the permeated water vapor and oxygen reduces the level of electron injection into the OLED, which drastically decreases its performance.

Various permeation barrier materials and deposition methods for the passivating OTFTs and OLEDs have been investigated [5,6]. Among these,  $\text{SiO}_x\text{N}_y$  is one of the more suitable materials for a permeation barrier due to its diffusion barrier properties in addition to its high scratch hardness. Thin glassy coatings of optically

transparent silicon compounds such as  $\text{SiO}_2$ ,  $\text{SiO}_x\text{N}_y$ , and  $\text{Si}_3\text{N}_4$  can be deposited using plasma enhanced chemical vapor deposition (PECVD) [7,8]. PECVD is one of the techniques that allows industrial scale deposition of high-quality barrier coatings with good uniformity, good adhesion to the substrate, and the flexibility in various chemical compositions at a low temperature [9].

Inorganic dielectric thin films are generally brittle and can withstand only a limited amount of strain. Therefore, brittleness of the deposited inorganic thin films may limit reliability of the permeation barrier. An external tensile stress to an inorganic thin film will cause the formation of cracks, which can peel off from the substrate along the interface. One way to further improve the barrier performance is to form a multilayer thin film consisting of multiple layers of organic/inorganic thin films by alternatively depositing an organic material for smoothing and defect decoupling on the top of a thin inorganic material.

In this study,  $\text{SiO}_x\text{N}_y$  thin films were deposited at a low temperature by inductively coupled PECVD using various hexamethyldisilazane (HMDS,  $\text{Si}_2\text{NH}(\text{CH}_3)_6$ )/ $\text{NH}_3/\text{O}_2/\text{Ar}$  gas mixtures. The properties of  $\text{SiO}_x\text{N}_y$  thin films as a diffusion barrier against water permeation were examined by forming a multilayer thin film consisting of  $\text{SiO}_x\text{N}_y$ /parylene on PES.

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Table 1  
Process parameters used in the deposition of  $\text{SiO}_x\text{N}_y$  in this experiment

|                              |  |
|------------------------------|--|
| Substrate                    | Si wafer, PES  |
| Gas chemistry                | HMDS+Ar+O <sub>2</sub> +NH <sub>3</sub><br>O <sub>2</sub> +NH <sub>3</sub> =20 sccm, R=O <sub>2</sub> :NH <sub>3</sub> |
| Deposition temperature       | Room temperature   |
| Source power                 | 300 W  |
| DC bias voltage              | -150 V   |
| Bubbler temperature          | 50 °C  |
| Delivery line temperature    | 80 °C  |
| Flow rate of HMDS            | 6–14 sccm  |
| Flow rate of O <sub>2</sub>  | 2–18 sccm  |
| Flow rate of Ar              | 75 sccm  |
| Flow rate of NH <sub>3</sub> | 2–18 sccm  |

HMDS with a purity of 99.9% was used as the Si precursor because it is relatively safe, non-corrosive, and has been widely used in the integrated circuit industry as a photoresist adhesion promoting agent [10]. Parylene, which has excellent chemical stability, optical transmittance, and step coverage, was used as the organic material [11].

## 2. Experimental

For the deposition, a planar-type inductively coupled plasma (ICP) source made of a 3-turn copper coil and operated at 300 W of 13.56 MHz was used to deposit the  $\text{SiO}_x\text{N}_y$ . The substrate was maintained at a temperature <40 °C using a chiller and was biased at -150 V using a separate 13.56 MHz rf power source. The silicon precursor was HMDS, which was provided by Sigma-Aldrich Co. with a high purity of 99.9%. In addition to HMDS, NH<sub>3</sub>, O<sub>2</sub>, and Ar were used to deposit the  $\text{SiO}_x\text{N}_y$ . NH<sub>3</sub> was used to promote nitride formation, O<sub>2</sub> was used to increase the level of oxidation, and Ar was used to increase the rate of dissociation of the precursor molecules. In order to optimize the  $\text{SiO}_x\text{N}_y$  film, the HMDS flow rate was varied from 6 to 14 sccm or the O<sub>2</sub>:NH<sub>3</sub> ratio was varied at a total NH<sub>3</sub>+O<sub>2</sub> flow rate of 20 sccm. The Ar flow rate was maintained at 75 sccm. Table 1 shows the operating conditions for depositing the  $\text{SiO}_x\text{N}_y$  films.  $\text{SiO}_x\text{N}_y$  was deposited on a silicon wafer in order to measure the deposition rate and to characterize the deposited film. The films were also deposited on the 200- $\mu\text{m}$ -thick polyethersulfone (PES) in order to measure the water vapor transmission rate (WVTR). The parylene film, which was used for the formation of a multilayer thin film composed of parylene/ $\text{SiO}_x\text{N}_y$ , was deposited using a parylene coater (SCS, Inc., PDS 2010 LABCOTER) at a pressure of 20 mTorr and at room temperature.

The thickness of the deposited films was measured using a step profilometer (Tencor Inc. Alpha-step 500). The chemical composition and bonding states of the deposited  $\text{SiO}_x\text{N}_y$  films were investigated using X-ray photoelectron spectroscopy (XPS, VG Microtech Inc., ESCA2000) and Fourier transform infrared spectroscopy (FT-IR, Bruker IFS-66/S, Bruker), respectively. The functionality of the films was measured by FTIR on 300-nm-thick  $\text{SiO}_x\text{N}_y$ . The WVTR of the multilayer thin films on PES was measured using a WVTR measurement system (MOCON Inc., PERMATRAN-W Model3/33) at a relative humidity of 100%, temperature of 37.8 °C, and a N<sub>2</sub> flow rate of 6.4 sccm.

## 3. Results and discussion

Fig. 1 shows the deposition rate of  $\text{SiO}_x\text{N}_y$  thin films as a function of the gas HMDS flow rate at a O<sub>2</sub>:NH<sub>3</sub> ratio of 1:1 (Fig. 1(a)) and as a function of the O<sub>2</sub>:NH<sub>3</sub> ratio with the HMDS flow rate of 6 sccm (Fig. 1(b)). The total flow rate of Ar+O<sub>2</sub>+NH<sub>3</sub> was kept at 95 sccm and the operating pressure was maintained 21.33 pa. As shown in Fig. 1(a), the deposition rate increased from 68.2 nm/min to 374.5 nm/min with increasing HMDS flow rate from 6 to 14 sccm at an O<sub>2</sub>:NH<sub>3</sub> ratio of 1:1. The increase in the deposition rate with increasing HMDS flow rate appeared to be accompanied by an increase in the concentration of impurities such as -CH<sub>x</sub> in the film. Therefore, the film became darker and softer. As shown in the Fig. 1(b), an increase in the O<sub>2</sub>:NH<sub>3</sub> ratio from 1:9 to 9:1 at a HMDS flow rate of 6 sccm initially resulted in a slight increase in the deposition rate from 65.3 nm/min to 68.2 nm/min until the ratio reached 1:1. However, the deposition rate decreased to 57.1 nm/min with further increases to 9:1. There was an increase in  $\text{SiO}_x\text{N}_y$  formation with increasing oxygen ratio in the gas mixture while there was a corresponding decrease in the amount of organic

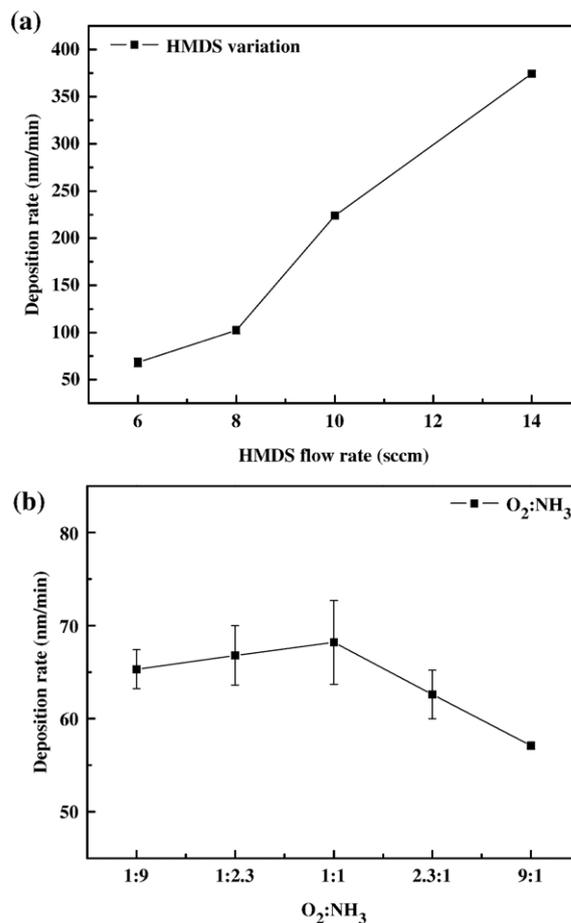


Fig. 1. Deposition rate of  $\text{SiO}_x\text{N}_y$  films measured (a) as a function of the HMDS flow rate for the ratio of 1:1 (b) and as a function of the oxygen ratio (O<sub>2</sub>:NH<sub>3</sub>) in the HMDS/Ar/O<sub>2</sub>/NH<sub>3</sub> gas mixture at a 99.9% HMDS flow rate of 6 sccm. Deposition conditions: (HMDS 6 sccm/(O<sub>2</sub>+NH<sub>3</sub>):20 sccm/Ar 75 sccm), 300 W of rf power, and -150 V of dc bias voltage, and 40 °C of substrate temperature.

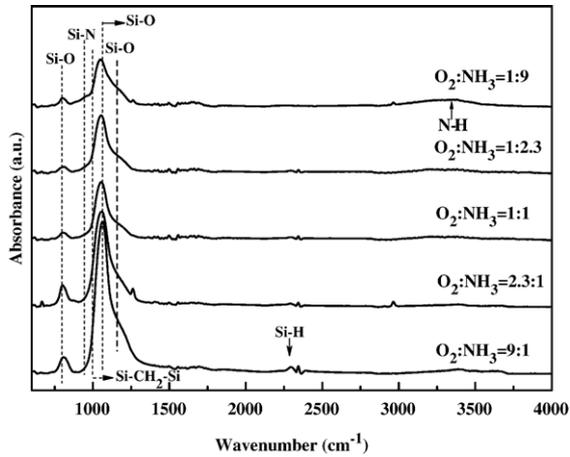


Fig. 2. FTIR spectra of the  $\text{SiO}_x\text{N}_y$  films as a function of the  $\text{O}_2:\text{NH}_3$  ratio. The deposition conditions are the same as those shown in Fig. 1.

impurities such as  $-\text{CH}_x$  in the film as a result of the removal of carbon by the formation of  $\text{CO}_x$ . Therefore, the initial slight increase in deposition rate with the increasing ratio appears to be related to the increased formation of  $\text{SiO}_x\text{N}_y$  from the vapor phase while the decrease in the deposition rate at the higher ratio appears to be related to the decrease in the amount of organic components in the film.

Fig. 2 shows the FTIR spectra indicating the chemical binding states of the deposited films as a function of the  $\text{O}_2:\text{NH}_3$  ratio shown in Fig. 1. The figure shows that the film deposited with a ratio of 1:9 showed a broad peak approximately  $1020$  ( $\sim 1030$ )  $\text{cm}^{-1}$  related to  $\text{Si}-\text{CH}_2-\text{Si}$  bonding mixed with small peaks related to  $\text{Si}-\text{N}$  bonding approximately  $900$   $\text{cm}^{-1}$  and  $\text{Si}-\text{O}$  bonding approximately  $1070$   $\text{cm}^{-1}$ . In addition, the peaks at approximately  $3363$  ( $\sim 3373$ )  $\text{cm}^{-1}$  and  $800.4$   $\text{cm}^{-1}$  were assigned to  $\text{N}-\text{H}$  and  $\text{Si}-\text{O}$  bonding, respectively [12–14]. As shown in the figure, the peak heights related to  $\text{Si}-\text{CH}_2-\text{Si}$  and  $\text{N}-\text{H}$  bonding decreased with increasing  $\text{O}_2:\text{NH}_3$  ratio, while the peak heights related to  $\text{Si}-\text{O}$  bonding increased. Therefore, there was a decrease in the number of impurity binding states in the  $\text{Si}-\text{O}$  vibration in the deposited film with increasing the  $\text{O}_2:\text{NH}_3$  ratio. In addition, there was a decrease in the number of  $\text{Si}-\text{N}$ -type bonds with increasing  $\text{O}_2:\text{NH}_3$  ratio. Therefore, the film became more oxide-like  $\text{SiO}_x\text{N}_y$  with increasing  $\text{O}_2:\text{NH}_3$  ratio.

Table 2 shows the XPS binding energy of the elements in the  $\text{SiO}_x\text{N}_y$  films deposited as a function of the  $\text{O}_2:\text{NH}_3$  ratio.

Table 2  
Atomic concentration of  $\text{SiO}_x\text{N}_y$  films measured by XPS as a function of the  $\text{O}_2$  ratio in  $\text{O}_2:\text{NH}_3$

| $\text{O}_2:\text{NH}_3$ | Si (%) | C (%) | N (%) | O (%) |
|--------------------------|--------|-------|-------|-------|
| 1:9                      | 36.1   | 9.9   | 6     | 48    |
| 1:2.3                    | 39.5   | 4.4   | 4     | 52.1  |
| 1:1                      | 37.3   | 3.9   | 3.8   | 55    |
| 2.3:1                    | 38.9   | 1.2   | 2.2   | 57.7  |
| 9:1                      | 38.1   | 1.5   | 1     | 59.4  |

The deposition conditions are the same as those in Fig. 1.

Hydrogen could not be measured due to the detection limit of XPS. As shown in the table, an increase in the  $\text{O}_2:\text{NH}_3$  ratio did not alter the atomic percentage of Si significantly. However, an increase in the  $\text{O}_2:\text{NH}_3$  ratio from 1:9 to 9:1 increased the oxygen concentration significantly from 48% to 59.4% while decreasing the carbon concentration from 9.9% to 1.5% and the nitrogen concentration from 6% to 1% in the film. In a previous report, when the film was deposited with 5 sccm of 98% HMDS while keeping the other conditions constant, the carbon percentage remaining in the film was higher and decreased from 15% to 2% with increasing  $\text{O}_2:\text{NH}_3$  ratio from 1:9 to 9:1 (not shown). The lower carbon percentage obtained in this experiment appears to be related to the increase in the purity of HMDS.

Fig. 3 shows the XPS narrow scan data of  $\text{Si}_{2p}$  and  $\text{O}_{1s}$  for the  $\text{SiO}_x\text{N}_y$  film deposited at  $\text{O}_2:\text{NH}_3$  ratios ranging from 1:9 to 9:1. As shown in the figure, the binding of the Si core level increased from 103.3 eV to 103.7 eV with increasing  $\text{O}_2:\text{NH}_3$  ratio with a corresponding but slight increase in the binding energy of O from 532.8 eV to 533.1 eV. These peak locations are closely related to  $\text{Si}-\text{O}$  bonding, and an increase in the ratio changed the type of bonding to more  $\text{SiO}_2$ -like [15]. Therefore, a more oxide-like

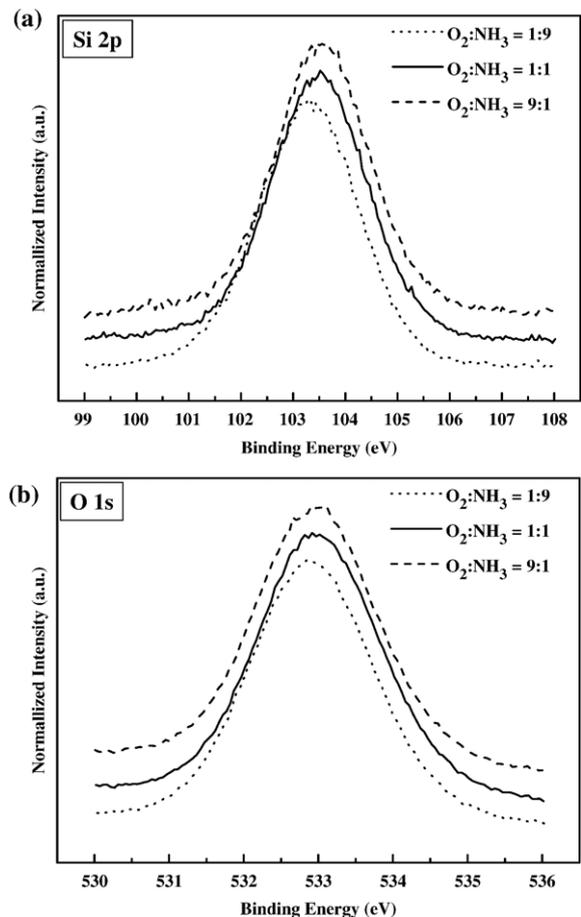


Fig. 3. XPS spectra of (a)  $\text{Si}_{2p}$  and (b)  $\text{O}_{1s}$  core levels of the  $\text{SiO}_x\text{N}_y$  thin films as a function of the  $\text{O}_2:\text{NH}_3$  ratio. The deposition conditions are the same as those shown in Fig. 1.

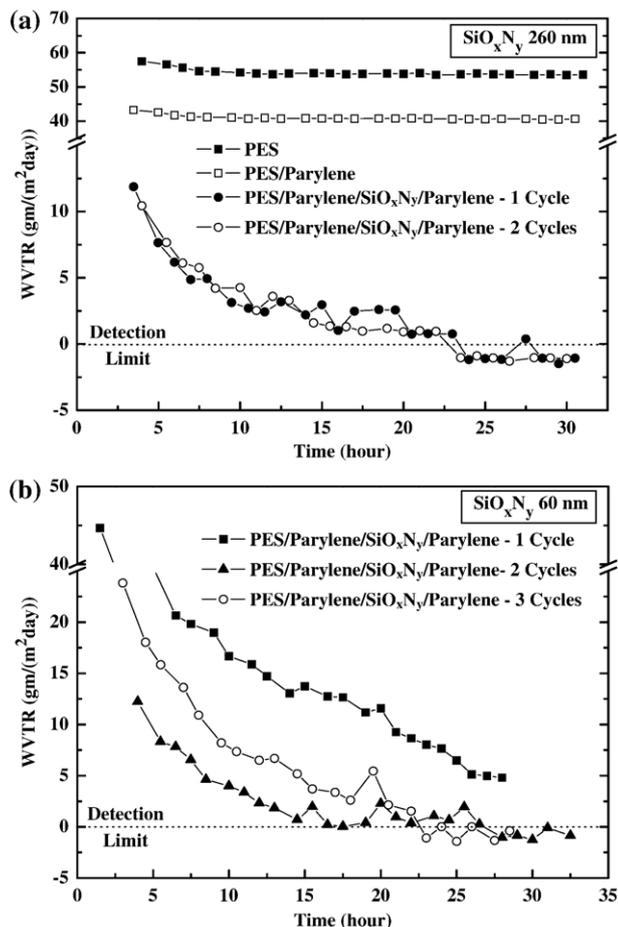


Fig. 4. WVTRs of multilayer thin film consisting of (a) one and two cycles of SiO<sub>x</sub>N<sub>y</sub> (260 nm)/parylene on PES (200  $\mu\text{m}$ )/parylene (1.2  $\mu\text{m}$ ). As references, the WVTR of 200- $\mu\text{m}$ -thick PES and PES (200  $\mu\text{m}$ )/parylene (800 nm) were also inserted. (b) One, two and three cycles of SiO<sub>x</sub>N<sub>y</sub> (60 nm)/parylene on PES (200  $\mu\text{m}$ )/parylene (1.2  $\mu\text{m}$ ). The deposition conditions used for SiO<sub>x</sub>N<sub>y</sub> deposition: HMDS (6 sccm)/O<sub>2</sub> (10 sccm)/NH<sub>3</sub> (10 sccm)/Ar (75 sccm), 300 W of the rf power, and -150 V dc bias voltage, and the substrate temperature 40 °C.

SiO<sub>x</sub>N<sub>y</sub> film could be obtained with increasing O<sub>2</sub>:NH<sub>3</sub> ratio, which is similar to the FTIR results shown in Fig. 2.

The film deposited at a O<sub>2</sub>:NH<sub>3</sub> ratio of 1:1 not only showed the highest deposition rate but the most SiO<sub>x</sub>N<sub>y</sub>-like characteristics with a hardness and transparency similar to the film deposited at a O<sub>2</sub>:NH<sub>3</sub> ratio of 9:1. Therefore, using the SiO<sub>x</sub>N<sub>y</sub> film deposited at a O<sub>2</sub>:NH<sub>3</sub> ratio of 1:1, a multilayer thin films composed of SiO<sub>x</sub>N<sub>y</sub>/parylene (1.2  $\mu\text{m}$ : on the top or 120 nm: between SiO<sub>x</sub>N<sub>y</sub> layers) was formed on the 200  $\mu\text{m}$  thick PES films and their WVTRs were measured. The structures of the multilayer film consisted of PES (200  $\mu\text{m}$ )/parylene (1.2  $\mu\text{m}$ )/SiO<sub>x</sub>N<sub>y</sub>/parylene (1.2  $\mu\text{m}$ )[1 cycle], PES (200  $\mu\text{m}$ )/parylene (1.2  $\mu\text{m}$ )/SiO<sub>x</sub>N<sub>y</sub>/parylene (1.2  $\mu\text{m}$ )/SiO<sub>x</sub>N<sub>y</sub>/parylene (1.2  $\mu\text{m}$ )[2 cycles], etc. Therefore, multiple cycle layers of SiO<sub>x</sub>N<sub>y</sub>/parylene were deposited on the PES (200  $\mu\text{m}$ )/parylene (1.2  $\mu\text{m}$ ). Two different SiO<sub>x</sub>N<sub>y</sub> thicknesses, 60 nm and 260 nm, were used and Fig. 4(a) shows the measured WVTRs of the multiple thin films deposited on the PES for a 260 nm-thick SiO<sub>x</sub>N<sub>y</sub> film. Fig. 4(b) shows the measured WVTRs of the multiple thin films deposited on the PES for a 60 nm-thick SiO<sub>x</sub>N<sub>y</sub> film. As references, the WVTRs of PES (200  $\mu\text{m}$ ) and PES (200  $\mu\text{m}$ )/

parylene (800 nm) obtained from a previous experiment [1] were also included. As shown in the figures, the WVTRs of the PES (200  $\mu\text{m}$ ) and PES (200  $\mu\text{m}$ )/parylene (800 nm) were 54.1  $\text{g}/(\text{m}^2 \text{day})$  and 40.4  $\text{g}/(\text{m}^2 \text{day})$ , respectively. Therefore, the deposition of an 800 nm thick parylene layer on the PES did not have any significant effect on blocking the permeation of water. Instead, the deposition of parylene is believed to be more effective in decreasing the surface roughness of the substrate, planarizing the substrate surface, etc. The deposition of 1 cycle SiO<sub>x</sub>N<sub>y</sub> (60 nm)/parylene (1.2  $\mu\text{m}$ ) on PES/parylene decreased the level of water vapor permeation significantly, as shown in Fig. 4(b). However, the WVTR was not enough to be applicable to devices such as OTFTs and OLEDs. A WVTR < 0.005  $\text{g}/(\text{m}^2 \text{day})$ , which is the detection limit of the measurement system, could be obtained by depositing a thicker SiO<sub>x</sub>N<sub>y</sub> layer, as shown in Fig. 4(a) for SiO<sub>x</sub>N<sub>y</sub> (260 nm)/parylene (1.2  $\mu\text{m}$ ) on PES/parylene. In addition, a WVTR < 0.005  $\text{g}/(\text{m}^2 \text{day})$  could be obtained for both 60-nm-thick SiO<sub>x</sub>N<sub>y</sub> and 260-nm-thick SiO<sub>x</sub>N<sub>y</sub> using a multiple layer thin film composed of more than 2 cycles of SiO<sub>x</sub>N<sub>y</sub>/parylene on PES/parylene.

#### 4. Conclusions

In this study, SiO<sub>x</sub>N<sub>y</sub> was deposited at low substrate temperatures using inductively coupled PECVD with HMDS (99.9%)/O<sub>2</sub>/Ar/NH<sub>3</sub>. The characteristics of these films as a water vapor permeation barrier material were investigated as a function of the O<sub>2</sub>:NH<sub>3</sub> ratio. In addition, the characteristics of the WVTR were investigated by forming multilayer thin films consisting of SiO<sub>x</sub>N<sub>y</sub>/parylene on PES. SiO<sub>x</sub>N<sub>y</sub> thin films with fewer impurities such as C and H could be deposited using a higher O<sub>2</sub>:NH<sub>3</sub> ratio. With increasing oxygen ratio, the Si–N bonds in the deposited film were also replaced by Si–O bonds. Therefore, SiO<sub>2</sub>-like SiO<sub>x</sub>N<sub>y</sub> could be obtained. The deposition of a one cycle 260-nm layer of SiO<sub>x</sub>N<sub>y</sub> (deposited with an oxygen ratio of 1:1)/parylene on PES (200  $\mu\text{m}$ )/parylene (1.2  $\mu\text{m}$ ) showed a WVTR 0.005  $\text{g}/(\text{m}^2 \text{day})$ , which is suitable to OTFTs and possibly to OLEDs. When the thickness of the SiO<sub>x</sub>N<sub>y</sub> was 60 nm, a WVTR < 0.005  $\text{g}/(\text{m}^2 \text{day})$  could be obtained using at least two cycles of SiO<sub>x</sub>N<sub>y</sub>/parylene layers on PES (200  $\mu\text{m}$ )/parylene (1.2  $\mu\text{m}$ ).

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