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Enhancement of barrier properties of aluminum oxide layer by optimization of plasma-enhanced atomic layer deposition process

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ABSTRACT

Aluminum oxide (Al_xO_y) layers were deposited on polyethylene naphthalate substrates by low frequency plasma-enhanced atomic layer deposition process for barrier property enhancement. Trimethylaluminum and oxygen plasma were used as precursor and reactant materials, respectively. In order to enhance the barrier properties several process parameters were examined such as plasma power, working pressure and electrode–substrate distance. Increase of plasma power enhanced the reactivity of activated atomic and molecular oxygen to reduce the carbon contents in Al_xO_y layer, which appeared to enhance the barrier properties. But too high power caused generation of byproducts which were reincorporated in Al_xO_y layer to reduce the barrier properties. Plasma generated at lower working pressure was provided with an additional energy for reactions and had more diffusion of the plasma. The O/Al ratio of the layer approached the stoichiometric value by increasing the electrode–substrate distance. At the following conditions: 300 W of plasma power, 26.7 Pa of working pressure and 50 mm of electrode–substrate distance, water vapor transmission rates of the Al_xO_y layer reached 8.85×10^{-4} g/m^2 day.

1. Introduction

Flexible display is regarded as a promising future display technology, and many efforts have been carried out for supplying the components for flexible display devices such as liquid crystal display, electrophoretic display and organic light emitting (OLED) devices. Plastic substrate with high barrier properties is one of the most important among the several components for flexible display devices. Especially, OLED is gaining much interest due to its several advantages over the other display devices [1]. However, the barrier property required for OLEDs is less than 1.0×10^{-5} g/m^2 day, because water and oxygen vapor can deteriorate the OLED performance in many ways, one of which is that water vapor can oxidize the metallic cathode, which reduces the electron injection in OLED device [2,3]. Thus, it is imperative for the plastic substrate to have barrier properties to prevent water and oxygen vapor permeation.

Single inorganic gas barrier layers, which are approximately 10–100 nm in thickness, are fabricated by various physical and chemical techniques such as sputtering [4], thermal evaporation [5], plasma enhanced chemical vapor deposition [6] and atomic layer deposition [7]. Among these techniques, atomic layer deposition (ALD) method is known to produce densely packed, virtually defect-free, highly uniform, conformal films, and can therefore be used to deposit high-quality single gas barrier layer [8,9]. ALD method is a process, in which alternating pulses of a precursor and a reactant produce one atomic layer at a time. Due to the characteristics of self-limiting and surface reactions, film thickness is only dependent on the number of process cycles providing extremely high uniformity and thickness control. Therefore, ALD method has gained significant interest for the deposition of thin oxide layer with good barrier properties [10].

Aluminum oxide (Al_xO_y) layers were grown using trimethylaluminum (TMA, $Al(CH_3)_3$) and H_2O vapor as precursor and oxygen source, respectively. However, typical ALD method using H_2O vapor as oxygen source has the limitation in the choice of precursor chemistry and that results in a narrow process window. Additionally, it was reported that ALD Al_xO_y layers had unwanted inhomogeneous phase, oxygen-deficient layer, at the incipient stage when the Al_xO_y layers were grown using TMA and H_2O vapor [11]. Recently, ALD processes in which O_3 and O_2 plasma are used as alternative oxygen sources instead of H_2O vapor are increasingly gaining more attention, because of potential advantages such as improved film quality, increased flexibility in process conditions, and feasibility of lower deposition temperatures [12]. When O_2 plasma is used, chemical activation of the oxidant has already occurred in the gas phase and, therefore, the use of a lower deposition temperature is in many cases facilitated [13]. Goldstein et al. investigated the effect of deposition temperature on the surface chemical property of Al_xO_y layers deposited via ALD, comparing the effects of H_2O vapor and O_3 as oxygen source [14]. Ha et al. investigated the influence of oxygen source, such as H_2O vapor, O_2 plasma and O_3, on the electrical and interfacial properties with the function of layer thickness of Al_xO_y [15].

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Although there have been extensive studies of gas barrier layers using plasma-enhanced atomic layer deposition (PEALD) process, most of the previous reports were focused on the high frequency (13.56 MHz) [16] or microwave (2.56 GHz) plasma [17]. In our previous work, we have demonstrated that aluminum oxide layers were deposited by PEALD process at low frequency (60 Hz), and that we have achieved low water vapor transmission rates (WVTR) of 4.0×10^{-3} g/m^2 day at 38 °C and 100% relative humidity (RH) for several plastic substrates such as polyethersulfone, polycarbonate and polyethylene naphthalate (PEN) [18]. Barrier properties should be more improved for its potential application to OLED device. In this work, Al_xO_y barrier layer was deposited on PEN substrates by low frequency PEALD using TMA as precursor and O_2 plasma as oxygen source. For its application to flexible display devices, especially to OLED device, we still need to enhance the barrier properties to the level below 10^{-3} g/m^2 day. We have investigated the influence of plasma parameters including plasma power, working pressure and electrode–substrate distance, on the barrier property of Al_xO_y layers. We were able to achieve WVTR values down to the order of 10^{-4} g/m^2 day.

2. Experimental details

Al_xO_y layers were deposited on PEN (Q65, DuPont-Teijin Films) substrates by using low frequency PEALD apparatus as used in previous work [18]. This reaction chamber was 203.2 mm tall with 457.2 mm diameter and equipped with a load-locked preparation chamber. The reaction chamber could accommodate substrates of 150 mm square size for uniformity. PEALD reactor was installed in a clean room of Class 10,000 and was kept clean. Especially, the inside of the reactor chamber was maintained free from dust and residual contamination by periodical cleaning.

Prior to deposition, PEN substrates with a thickness of 125 μm were cut into 50 × 50 mm pieces, and cleaning of the substrates was carried out for 10 min in isopropyl alcohol ultrasonication chamber in a clean room. Then, the substrates were sufficiently dried at 80 °C in an oven. Plastic substrate should be fixed for quick loading into the load-lock chamber, and it was lightly taped on slide glass by applying the double-sided tape at the edge of the substrate. Slide glass cleaning and taping process were also conducted in a clean room to minimize the contamination. After PEALD deposition, the sample was easily removed from the glass without any damage. Only the central part (30 × 30 mm) out of 50 × 50 mm sample was used for WVTR measurement to avoid any edge taping problem. When the substrates were transferred to the deposition system they were heated at 80 °C for 60 min in the reaction chamber at a base pressure of 0.1 Pa to remove residual water in the substrate.

Pure argon (purity: 99.999%) gas was blown through the reactor at 500 sccm and produced a pressure of 133.3 Pa. Also, Ar gas was used as the carrier gas for the TMA vapor as well as the purge gas. TMA precursor was kept at 20 °C during the entire deposition process, and all Al_xO_y samples were deposited at a substrate temperature of 120 °C. Al_xO_y layer was deposited by alternating supplies of TMA and oxygen into the carrier gas. One cycle of PEALD process consisted of a 1.0 s TMA pulse, a 10 s purge, a 5.0 s oxygen pulse and another 10 s purge. The optimization of substrate temperature and purge time has been described in detail in our previous work [18]. The electrodes are capacitively coupled with a low frequency (60 Hz) plasma source, and RF plasma pulse was applied for 3.0 s to produce oxygen radicals during the injection of oxygen gas. Plasma power ranged from 100 to 700 W and 100 cycles were applied for each sample. The working pressure was in the range of 26.7 to 133.3 Pa, and the electrode–substrate distance was varied from 20 to 60 mm.

WVTR values were determined by using PERMATRAN-W Model 3/33 (MOCON Inc., USA) for values greater than the detection limit of the apparatus, 4.0×10^{-3} g/m^2 day at 38 °C and 100% RH. WVTR values below the detection limit of MOCON apparatus were determined by calcium cell test by monitoring the change in ohmic behavior of the calcium cell layer [19]. Calcium cell was aged in a climatic chamber at 38 °C and 90% RH. The thickness and refractive index of layer were measured by using a spectroscopic ellipsometer (V-VASE, J.A. Woollam Co., Inc., USA). The chemical composition of the layer was analyzed using X-ray photoelectron spectroscopy (XPS) with a K-Alpha system (Thermo Scientific, UK) and Al Kα X-ray source was used with a micro-focused monochromator. The base pressure of the ultrahigh vacuum chamber was lower than 4×10^{-9} Pa, and the resolution was 0.9 eV/104 counts per second. Atomic force microscope (AFM, Digital Instruments Nanoscope III) was utilized to analyze the surface morphology of the layer via tapping mode using nanosensor silicon tips with the resonance frequency of 130 kHz.

3. Results and discussion

In Fig. 1, variation of thickness of Al_xO_y layer was examined as a function of the number of cycles. Thickness was proportional to the number of process cycles, and thereby growth rate was kept constant. In ALD process, the growth rate of layer depends more on the number of process cycles rather than the cycle time or the intensity of the material pulse, since growth mechanism of ALD was dominated by self-limiting surface adsorption and reaction [20]. The growth rate was determined as the total layer thickness divided by the number of process cycles, and was found to be 0.24 nm/cycle, which is greater than that of the ALD process using H_2O as reactant material [7]. Oxygen radicals from O_2 plasma had higher reactivity than that from H_2O vapor, and they would attack TMA more aggressively to produce much more fragments of precursors [15].

For the study of the plasma parameters, TMA pulse time, oxygen pulse time, purge time and plasma exposure time were set at 1.0 s, 5.0 s, 10 s and 3 s, respectively. Effects of plasma parameters, such as plasma power, working pressure and electrode–substrate distance, were examined. Thickness of Al_xO_y layer should be optimized in terms of performance as well as energy consumption. It was confirmed from the preliminary experiments that there were no more reductions of WVTR values after the hundred cycles. Reproducible barrier properties were obtained at the hundred cycles which made 24 nm thickness. Based on these results thickness was kept as 24 nm in this work.

As a first plasma parameter in the deposition of Al_xO_y layer, plasma power was varied from 100 to 700 W. The working pressure and substrate temperature were maintained constant at 133.3 Pa and 120 °C, respectively, and electrode–substrate distance was fixed at 20 mm. As shown in Fig. 2, the carbon atomic concentration of layer has decreased from 8.41% to 1.42%, when plasma power increased from 100 W to 300 W. The increase of plasma power enhanced the reactivity of O_2 plasma which efficiently removes the trimethyl group from TMA source and increases reaction of TMA source and activated oxygen in the plasma process during deposition [21], which resulted in the decrease of

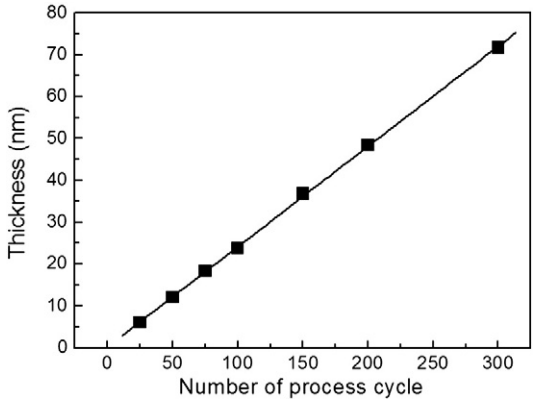

Fig. 1. Thickness of Al_xO_y layers as a function of the number of cycles.

carbon atomic concentration. However, carbon atomic concentration was slightly increased from 3.87% to 4.39% by increasing the plasma power from 500 W to 700 W, because it is believed that CH_x byproducts were generated by decomposition of TMA at excess plasma power and reincorporated in Al_xO_y layer [22]. Increase of plasma power from 100 to 300 W also resulted in an increase in the refractive index of Al_xO_y layer from 1.614 to 1.706, which may indicate an increase in film density. Refractive index began to slightly decrease beyond 300 W which may be due to the incorporation of the CH_x byproducts formed at high power [23]. The refractive index in general depends on molecular weight and molecular polarizability as well as density. Among this, the density is a primary determinant of the refractive index because the refractive index results from the collective response of electric dipoles excited by the external applied field and the number of dipoles in a given volume is closely related to the density [24]. Also, it was reported that the refractive index of impurity-free Al_2O_3 was 1.767 [25] and the decrease of refractive index was mainly caused by the incorporation of impurities such as carbon [26]. Fig. 3 shows the variation of WVTR values for the samples made using varying plasma power. PEN substrate with Al_xO_y layer deposited by 100 cycles reached the WVTR value of 3.12×10^{-3} g/m^2 day, which represents significant reduction of WVTR by three orders of magnitude when compared with that of bare PEN substrate (1.12 g/m^2 day). The improvement of barrier property of layers could be attributed to the dense and conformal characteristics of Al_xO_y layers by PEALD process. The best plasma power condition based on these results is determined to be 300 W for subsequent experiments.

Fig. 4 shows the carbon atomic concentration and refractive index of Al_xO_y layers deposited at different working pressures ranging from 26.7 to 133.3 Pa at plasma power of 300 W and electrode–substrate distance of 20 mm. As the working pressure decreased, the carbon atomic concentration was decreased and the refractive index increased. At working pressure of 26.7 Pa, the carbon atomic concentration and refractive index of layer showed the values of 0.47% and 1.721, respectively. However, there were no significant changes for thickness of layers with working pressure. Plasma generated at lower working pressure was provided with an additional energy for reactions at the film surface and could improve the film density. Former investigations showed that at low pressure more diffusion of the plasma radicals was achieved, and the plasma was more active than that at high working pressure [27]. With increasing working pressure, the plasma was more confined to the source region and resulted in less diffusion to downstream. Then the electron temperature decreased to result in the loss of electrons and ions by diffusion. Fig. 5 shows that WVTR value decreased as the working pressure decreased as expected and it reached the value of 1.11×10^{-3} g/m^2 day at lower working pressure of 26.7 Pa. In this case calcium cell test method was employed, since the WVTR values exceeded the detection limit of MOCON PERMATRAN apparatus. At least three samples were tested to make reproducible data. In Fig. 6, root mean square (RMS) values from AFM images were determined to examine the effects of working pressure on surface roughness of Al_xO_y layers deposited on PEN substrate. In all samples, each measurement was done three times for three different positions in one sample to obtain a mean value of the RMS. The standard deviation of the different measurements was less than 0.1 nm, indicating that the results are reproducible. RMS value appears to decrease at lower working pressure.

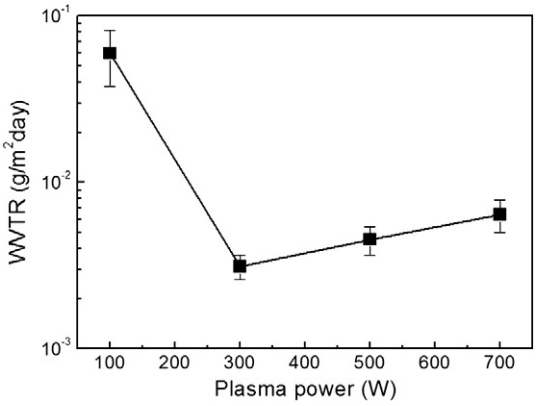

Fig. 3. WVTR values of Al_xO_y layers as a function of plasma power (WVTR below MOCON detection limit were measured again by calcium cell test at 38 °C and 90% RH).

Investigation of electrode–substrate distance was performed at plasma power of 300 W and working pressure of 26.7 Pa. Distance was varied from 20 to 60 mm and the XPS spectra of the Al 2p and the O 1s core levels at distances of 20 and 50 mm were compared in Fig. 7. The peak positions were calibrated using the binding energy of the adventitious C 1s signal (284.5 eV). In Fig. 7(a) Al $2p_A$ peak at ~74.3 eV is assigned to the Al(III) ions of oxide matrix, and the Al $2p_B$ peak at ~75.7 eV is assigned to the Al(III) ions of hydroxide matrix (likely AlOOH or $Al(OH)_3$ species) [28]. In Fig. 7(b), O $1s_A$ peak at ~530.7 eV is attributed to the oxide ions of the alumina matrix, and the O $1s_B$ peak at ~532.1 eV is attributed to hydroxyl groups or contaminants (carboxyls and/or carbonates) [23]. Relative intensities of Al $2p_A$ and Al $2p_B$ peaks were 84.4% and 15.6% at a distance of 20 mm and they were changed to 94.8% and 5.2% at a distance of 50 mm. Relative intensities of O $1s_A$ and O $1s_B$ peaks were 71.5% and 28.5% at a distance of 20 mm and they were changed to 82.3% and 17.7% at a distance of 50 mm. This result indicated that layers deposited at shorter distance have residual OH-related ligands due to their incomplete removal during deposition. The presence of AlOOH or $Al(OH)_3$ could deteriorate the barrier properties due to their low densities and poor O/Al ratio at shorter distance. It was reported that Fourier-transform infrared spectroscopy confirmed the accumulation

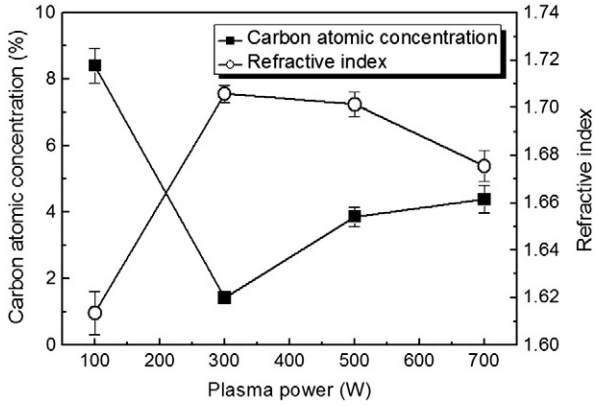

Fig. 2. Carbon atomic concentration and refractive index of Al_xO_y layers as a function of plasma power.

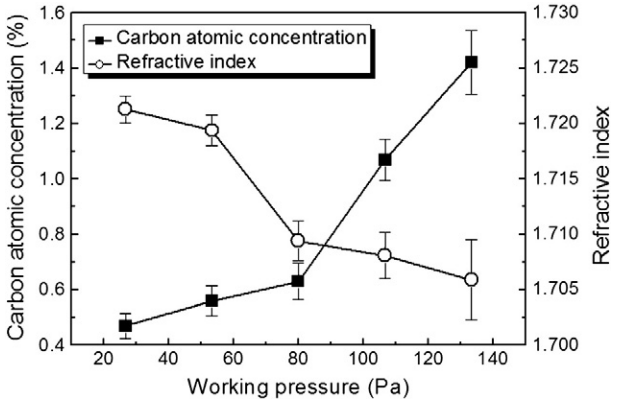

Fig. 4. Carbon atomic concentration and refractive index of Al_xO_y layers as a function of working pressure.

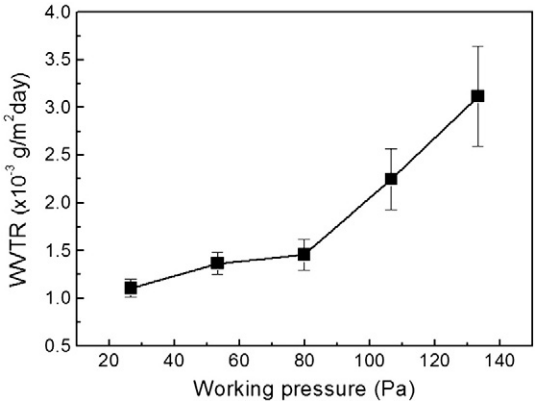

Fig. 5. WVTR of Al_2O_3 layers as a function of working pressure (WVTR were measured by calcium cell test at 38 °C and 90% RH).

of Al-OH species due to the incomplete precursor reaction in Al_xO_y layers deposition by ALD process below 177 °C [29]. Despite low substrate temperature (120 °C), we could obtain the Al_xO_y layer with low contents of hydroxide group (AlOOH or $Al(OH)_3$ species) using optimization of electrode–substrate distance. Fig. 8 shows the variations of O/Al ratio and WVTR with the increase of electrode–substrate distance. The O/Al ratio was estimated from the O $1s_A$ and Al $2p_A$ components [28]. O/Al ratio increased with distance up to 50 mm and reached the value of 1.46. Based on the stoichiometry of Al_xO_y formation the ideal O/Al ratio should be 1.5 and the value obtained in this work was quite close to the ideal value. WVTR value decreased gradually when the distance increased up to 50 mm, and it reached the minimum value, 8.85×10^{-4} g/m^2 day at 50 mm, which was the best value in this work. When the distance increased to 60 mm, O/Al ratio suddenly dropped and WVTR increased drastically to 1.63×10^{-3} g/m^2 day. The distance which is too long may have resulted in the reduction of the concentration of O radicals present for the reactions due to their limited lifetime, which may then have resulted in less complete reaction between TMA and O radicals. O/Al ratio decrease at 60 mm distance increased the impurities which act as defects of Al_xO_y layer. Consequently, the WVTR value of Al_xO_y layer deteriorated. It is important to control the electrode–substrate distance to sustain stable plasma that is used to deposit high-quality Al_xO_y layer [30]. Even though the process in this work was performed at a temperature of 120 °C, which is a relatively lower temperature than of the other works, it achieved excellent WVTR value at the studied conditions.

4. Conclusion

Plasma power affected the reactivity of activated atomic and molecular oxygen, which we believe efficiently removes the trimethyl group from TMA source in the plasma process during deposition. Increase of plasma power appears to have enhanced the density and refractive index of the layer and best WVTR values were obtained at 300 W. Excess power beyond 300 W is believed to have caused the formation of CH_x byproducts from decomposition of TMA and they were incorporated in the layer to increase the carbon content of the layer. Lowering the working pressure may have provided additional energy of the plasma radicals for reactions at the film surface and enhanced their diffusion. Varying the electrode–substrate distance appears to have affected the radical lifetime of plasma during deposition. Consequently, the carbon atomic concentration of the layer deposited at electrode–substrate distance of 50 mm approached the ideal O/Al ratio to attain more complete precursor reaction. The best WVTR value obtained as a result of this study at 300 W, low working pressure and electrode-to-substrate distance was 8.85×10^{-4} g/m^2 day. This is a promising result especially for single layer barrier films on polymer substrates.

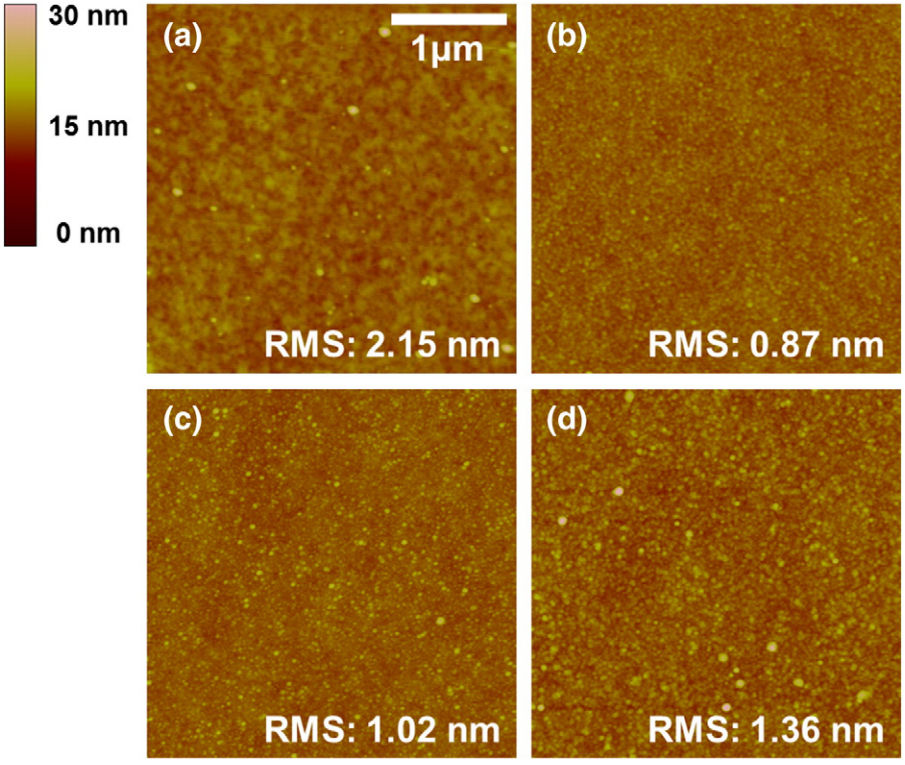

Fig. 6. AFM image of Al_xO_y layers as a function of working pressure; (a) bare PEN, (b) 26.7 Pa, (c) 79.9 Pa, and (d) 133.3 Pa.

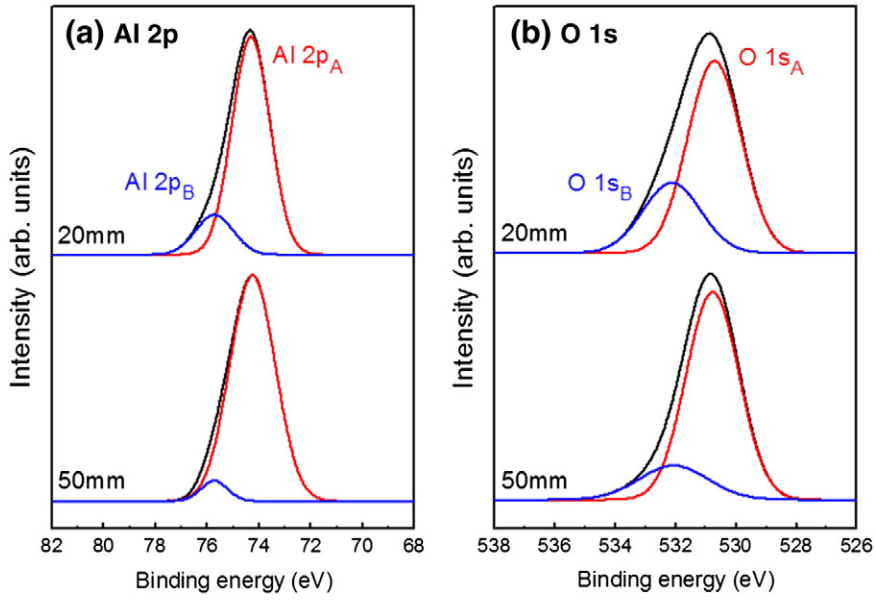

Fig. 7. XPS spectra of Al_xO_y layers as a function of electrode–substrate distance; (a) Al 2p peak and (b) O 1s peak.

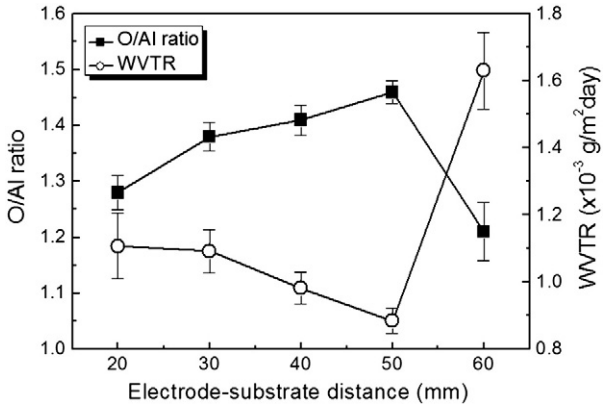

Fig. 8. O/Al ratio and WVTR values of Al_xO_y layers as a function of electrode–substrate distance; the O/Al ratio calculated with O 1s$_A$ and Al 2p$_A$ components (WVTR were measured by calcium cell test at 38 °C and 90% RH).

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