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Effect of bias frequency variation on the characteristics of SiO_X thin films deposited by atmospheric pressure chemical vapor deposition using a double discharge system

Jaebeom Park [a], Ga Young Kim [b], Geunyoung Yeom [a,b,*]

[a] SKKU Advanced Institute of Nano Technology (SAINT), Sungkyunkwan University, Suwon, Kyunggi-do 440-746, South Korea
[b] Department of Materials Science and Engineering, Sungkyunkwan University, Suwon, Kyunggi-do 440-746, South Korea

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ABSTRACT

SiO_x film was deposited with a double discharge system composed of a source discharge and a direct-type discharge (bias discharge) using a gas mixture of hexamethyldisilazane (400 sccm)/O_2 (20 slm)/He (5 slm)/Ar (10 slm) to act as a thin film passivation layer. Especially, the effect of the quasi-pulse power frequency for the substrate biasing on the plasma characteristics and characteristics of SiO_x was investigated. As the frequency of the bias discharge was increased, the deposition rate of SiO_x increased from 41.4 nm/cycle (cycle is one pass of the substrate in the in-line plasma source and, here, nm/cycle corresponds to nm/min) (20 kHz) to 137.1 nm/cycle (60 kHz), the mechanical hardness increased from 7.4 GPa (20 kHz) to 8.5 GPa (60 kHz), the roughness of the SiO_x surface decreased from 6.35 nm (20 kHz) to 1.37 nm (50 kHz) and the amount of carbon and nitrogen impurities decreased to less than 1%.The improvement of the deposited SiO_x thin film characteristics was related to the increased plasma density, greater gas dissociation and higher bombarding ion flux to the substrate caused by the increase of the power consumption, while maintaining the ion bombardment energy with increasing the bias frequency.

1. Introduction

Much attention has been paid to organic material-based devices such as organic light-emitting diodes (OLEDs) and organic thin film transistors (OTFTs), due to their low temperature and low-cost fabrication process and potential application to large-area flexible electronics, etc. However, the performance of organic material-based devices degrades easily and the device lifetime is also significantly limited by the ambient conditions [1–4]. Therefore, the passivation of organic material-based devices is necessary to extend the device performance and lifetime by protecting the organic active layers from ambient air. To protect the flexible display devices from the atmosphere, a thin film permeation barrier composed of a single inorganic layer or a multilayer should be formed both on the flexible substrates and on the surface of the electronic devices, as studied by various researchers [5–7]. Especially, the deposition of low temperature inorganic material films on OTFTs and OLEDs has been studied more intensively, because it is the most important material for preventing the diffusion of oxidizing gases[8–10]. Various physical and chemical thin film deposition methods, such as low pressure plasma enhanced chemical vapor deposition (PECVD) [11,12], atomic layer deposition[13–16], sputter deposition[17], atmospheric pressure PECVD (AP-PECVD)[18,19], etc., have been investigated.

Among the various techniques studied for the deposition of low temperature inorganic thin films on flexible polymer substrates, AP-PECVD has been investigated as one of the methods that could deposit materials at a low cost of ownership, due to its ability to perform processing without an expensive vacuum processing system and in-line processing or roll-to-toll processing, due to the lack of need for a loadlock system. Especially, among the various AP-PECVD techniques[20], dielectric barrier discharges (DBDs) which are composed of two parallel electrodes having a dielectric barrier material on one or both electrode surfaces have been widely investigated, due to the easier generation of uniform glow discharges over a large substrate area [21]. Generally, DBD is used as a capacitively coupled direct-type discharge configuration by applying alternating current (AC), radio-frequency, or pulse-type power to one electrode, while the substrate is located on the facing ground electrode. However, when DBDs are used in the deposition of SiO_x using various silicon containing gas mixtures with oxygen, the substrate and electronic devices can be easily damaged by the formation of a filamentary discharge, especially under the high power conditions required to achieve a high deposition rate or with a high oxygen content to deposit more stoichiometric SiO_x [22–24]. To prevent damage to the substrate and devices caused by the filamentary discharges, in our previous research [25] a remote-type AP-PECVD was used by locating the substrate separately from the DBD source on a third electrode located below the DBD source during the deposition of the SiO_x thin film. However, this system has other problems, such as a lower deposition rate, porous film properties, etc. Therefore, inorganic thin films, which are deposited by remote-type AP-PECVDs, are not suitable for use as a

* Corresponding author at: SKKU Advanced Institute of Nano Technology (SAINT), Sungkyunkwan University, Suwon, Kyunggi-do 440-746, South Korea. Tel.: +82 31 290 7395 Fax: +82 31 299 6565.
E-mail address: gyyeom@skku.edu (G. Yeom).

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passivation layer, due to the inappropriate characteristics of the inorganic layer.

Previously, a double DBD system composed of a source discharge and a direct-type DBD (bias discharge) was used for the deposition of SiO_x at a low temperature to investigate the possibility of depositing SiO_x at a higher rate without forming a filamentary discharge. The result showed that, due to the formation of a direct-type DBD on the substrate electrode using an AC power source, in addition to the source discharge system using another AC power source, the SiO_x thin films were deposited at a high deposition rate without forming a filamentary discharge and showed improved physical and chemical properties which are close to those of SiO_2 [26,27].

G.H. Kim et al.[28] studied the effect of the operating frequency on the power consumption in the plasma during the operation of a DBD and found that, because the amount of surface charge on the electrode is proportional to the capacitance of the DBD and to the discharge period, the maximum consumed power dissipation of a DBD can be achieved at a specific operating frequency, which may depend on the capacitance of the reactor[28,29]. Therefore, in this study, the effects of the operating frequency on the double discharge system and the SiO_x deposition process were studied to obtain a higher power dissipation efficiency. Especially, to further study the effect of the direct-type DBD, that is, the substrate biasing, in addition to the source discharge system, on the characteristics of the deposited SiO_x film, a quasi pulse-type power was used for the direct-type DBD (substrate biasing), because a pulse-type power is more stable and tends to suppress the filamentary discharge [30,31]. Also, the frequency of the pulse-type bias power applied to the direct-type DBD was varied and its effects on the SiO_x thin film properties was investigated with a gas mixture composed of hexamethyldisilazane (HMDS)/O_2/Hes/Ar.

2. Experimental details

In this study, a modified AP-PECVD system, that is a double discharge system, is used for the deposition of SiO_x. The discharge system is composed of a source discharge system for gas dissociation and a direct-type DBD source for substrate biasing and is shown in Fig. 1. The source discharge system was located on the top portion of the system and consisted of three flat metal electrodes covered with a 3 mm-thick ceramic plate. The air gap between the electrodes was about 1.5 mm. The ground electrode with a size of 60 mm(width) × 130 mm(length) × 40 mm(height) was located at the center of the source discharge system, while the two powered electrodes with a size of 25 mm(width) × 130 mm(length) × 40 mm(height) were located on the outside of the ground electrode for the formation of two DBD systems. A separate electrode with a size of 113 mm(width) × 130 mm(length) × 20 mm(height) covered with a 1.0 mm-thick quartz plate was located 1.5 mm below the source discharge system for the formation of a direct-type DBD for substrate biasing, in addition to source discharge system for gas dissociation.

HMDS (Sigma-Aldrich Co, purity 99.9%, $Si_2NH(CH_3)_6$) was used as the precursor for SiO_x deposition and was delivered to the source using He as a carrier gas. As the substrate, silicon wafers were used and, to deposit the SiO_x thin films, a gas mixture composed of HMDS (400 sccm)/O_2 (20 slm)/He (5 slm)/Ar (10 slm) was used. In the gas mixture, He/Ar was used as the discharge gas and O_2 was used for the oxidation of HMDS. HMDS carried by He is fed from the bottom side of the source discharge system electrode while the gas mixture of O_2/Ar is fed downward from the top side of the source discharge system. 5–7 kV of 30 kHz AC power was applied to the two outside electrodes, while the center electrode is grounded for the formation of the source discharge system. On the substrate electrode located below the source discharge system, a quasi pulse-type power supply was connected and the frequency and voltage of the pulse power (20–60 kHz, 4–10 kV) were varied for the generation of a separate direct-type DBD for substrate biasing. The substrate temperature was maintained below 80 °C using a chiller. During the deposition, the silicon substrate located on the direct-type DBD electrode was moved at a speed of 0.3 m/min. As the deposition rate, nm/cycle was used because the substrate continuously moves under the plasma source. One cycle represents 1 pass under the plasma source and, as the deposition rate, cycle/min corresponds to nm/min because the width of the deposition source is 300 mm.

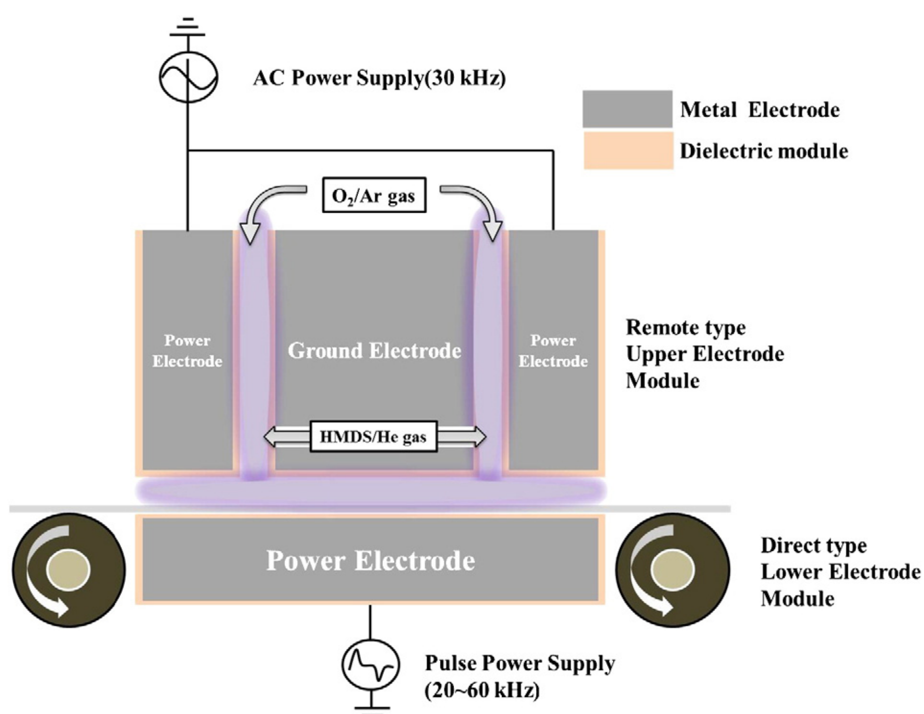

Fig. 1. In-line double discharge system used in the deposition of SiO_x thin film composed of a remote-type DBD above the substrate and a direct-type DBD on the substrate.

The thickness of the deposited SiO_x thin film was measured using a step profilometer (Tencor, Alpha step 500) and the chemical composition of the deposited SiO_x thin film was measured by X-ray photoelectron spectroscopy (XPS; Thermo Electronics, Multilab ESCA2000). For removing the surface oxidation, pre sputter was examined by XPS (ESCA2000, VG Microtech Inc.) using an Al*K*α twin-anode source with photon energy of 1486.6 eV. The Ar^+ ion energy and ion current were set to 2 kV and 2 μA, respectively. The XPS spectra were deconvoluted using a least squares fitting technique by the Avantage fitting program supplied by VG Microetch. The hardness of the films was measured at a normal load of 30 mN using a nanoindentation instrument (MTS, Nano-indenter II). The voltage and current of the atmospheric pressure plasmas were measured by using a high voltage probe (Tektronix P6015A) and a current probe (Pearson electronics 6600), with an oscilloscope (Tektronix TDS 340A). The surface roughness of the deposited SiO_x thin film was observed by atomic force microscopy (AFM; XE 100). The optical emission intensities of the species emitted from the plasma just above the substrate were measured using optical emission spectroscopy (OES, SC-Technology, PCM 420).

3. Results and discussion

Fig. 2 shows the effect of the frequency (20–60 kHz) and input voltage (4–10 kV) of the bias pulse power to the substrate electrode for the generation of a direct-type DBD on the deposition rate of the SiO_x thin film. On the source discharge system above the substrate surface, AC voltages of 30 kHz and 7 kV were applied, while flowing a gas mixture of HMDS(400 sccm)/He (5 slm)/O_2(20 slm)/Ar (10 slm). The substrate was moved at a speed of 0.3 m/min. As shown in Fig. 2, increasing the frequency and input voltage of the quasi pulse power applied to the substrate electrode increased the SiO_x deposition rate. For example, increasing the input voltage from 4 to 10 kV at a frequency of 60 kHz increased the deposition rate from 5.8 to 137.1 nm/cycle and increasing the frequency from 20 to 60 kHz at an input voltage of 10 kV increased the SiO_x deposition rate from 41.4 to 137.1 nm/cycle. The increase of the SiO_x deposition rate with increasing bias pulse power frequency and input voltage is believed to be related to the increased dissociation of the HMDS and oxygen molecules in the discharge.

Using XPS, the atomic percentages of the elements in the SiO_x deposited by the double discharge system, such as silicon, oxygen, carbon, and nitrogen, were measured as a function of the pulse power frequency of the direct-type DBD at AC input voltages of 5 and 7 kV for the source discharge system source and the results are shown in Fig. 3. The binding energy of each bond has been referenced by [32] The input voltage of the bias pulse power was maintained at 10 kV and was directly applied to the bias electrode. As shown in Fig. 3, the oxygen percentage slightly increased from 62 to 65% with increasing pulse power frequency from 20 to 60 kHz, while the silicon atomic percentage remained similar. The amount of carbon and nitrogen impurities such as carbon and nitrogen decreased significantly from 2.0–2.5 to 0.4–0.5% and from 1.2–1.5 to 0.6–0.8% with increasing frequency from 20 to 60 kHz, respectively. Increasing the AC voltage applied to the source discharge system from 5 to 7 kV under the same quasi pulse power conditions also increased the oxygen percentage slightly without changing the silicon percentage, even though the variation of the carbon percentage and nitrogen percentage with increasing AC voltage from 5 to 7 kV appeared to be in the error range.)

The presence of carbon and nitrogen in the deposited SiO_x thin film is due to the HMDS ($Si_2NH(CH_3)_6$) used as the silicon precursor [33]. The HMDS is introduced near the substrate surface from the bubbler with the He carrier gas. In the case of the source discharge system, the HMDS is dissociated and combines with the dissociated oxygen which is fed from the top side of the source discharge system. Through the recombination of silicon in the partially dissociated HMDS which may be previously deposited on the substrate with the oxygen atoms formed by the dissociation of oxygen molecules, SiO_x is deposited on the substrate. However, when the HMDS is not fully dissociated in the discharge, the carbon and nitrogen in the partially dissociated HMDS remain on the deposited SiO_x as impurities.

Therefore, the decrease in the amounts of carbon and nitrogen and/or the increase in the amount of oxygen in the deposited SiO_x observed in Fig. 3 with increasing frequency of the pulse power and AC voltage applied to the source discharge system is believed to be related to the increased dissociation of HMDS and oxygen molecules. In addition, the SiO_x deposited for use as a thin film passivation layer needs to have a high material density. Generally, carbon and nitrogen in the deposited SiO_x are in bonding states such as Si–CH_3 and Si–NH_3, respectively, due to the deposition of partially dissociated HMDS without complete oxidation. Also, it is known that bonds such as –CH_3 and –NH_3 decrease the material density [34] and make the film porous. Therefore, increasing the pulse power frequency to the direct-type DBD increased not only the SiO_x deposition rate, as shown in Fig. 2, but also improved the characteristics of the deposited SiO_x thin film, as shown in Fig. 3, by increasing the dissociation of HMDS and oxygen molecules near the substrate surface.

The increase in the degree of dissociation with increasing quasi pulse power frequency is related to the characteristics of the pulse power used in our experiment. Fig. 4 a) and b) show the voltage shapes of the quasi pulse power measured at 20 and 60 kHz,

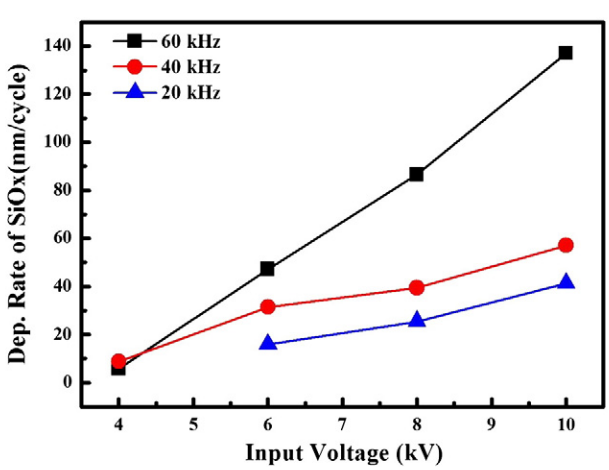

Fig. 2. Deposition rate of SiO_x thin film measured as a function of the voltage (4–10 kV) and frequency (20–60 kHz) of the pulse power to the substrate electrode. On the remote-type DBD above the substrate surface, an AC voltage of 7 kV at 30 kHz was applied, while flowing a gas mixture of HMDS(400 sccm)/He (5 slm)/O_2(20 slm)/Ar (10 slm). The speed of the substrate was fixed at 0.3 m/min.

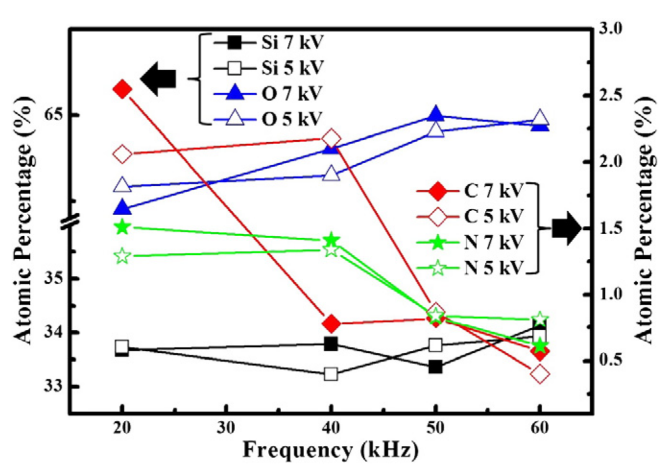

Fig. 3. Atomic percentages of Si, O, C, and N of the deposited SiO_x thin film investigated by XPS as a function of the pulse power frequency (20–60 kHz) of the direct-type DBD and the input voltage (5, 7 kV) of the remote-type DBD. The other deposition conditions are the same as those in Fig. 2.

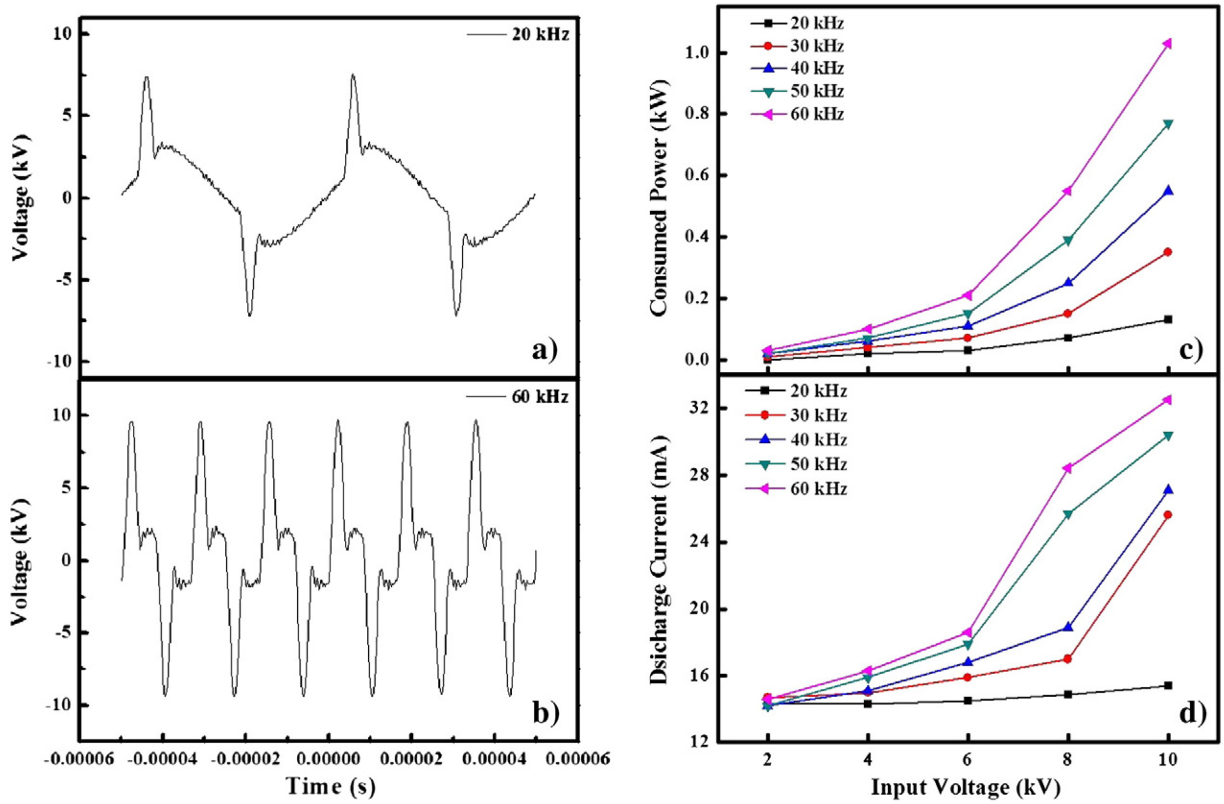

Fig. 4. The quasi pulse voltage curve on the substrate electrode measured at (a) 20 kHz and (b) 60 kHz. (c) Consumed power and (d) discharge current measured as functions of frequency (20–60 kHz) and input voltage (2–10 kV) of the pulse power to the substrate electrode. The other discharge conditions are the same as those in Fig. 1.

respectively, as a function of time for an input voltage of 10 kV, while applying an AC voltage of 7 kV at 30 kHz to the source discharge system. As shown in the figures, increasing the pulse power frequency increased the number of short pulses at a given time, because the duration of each pulse was constant at 4×10^{-6} s for all of the frequencies. Therefore, increasing the pulse power frequency increased the duty percentage. The calculated duty percentages at 20, 30, 40, 50, and 60 kHz were calculated to be 16, 24, 32, 40, and 48%, respectively. Due to the increase of the resulting plasma-on time with increasing pulse power frequency, increasing the quasi pulse power frequency increased the power input to the discharge at a given input voltage (that is, at the same peak voltage). Fig. 4 c) and d) show the consumed power and discharge current, respectively, measured as functions of the frequency and input voltage of the pulse power for the conditions shown in Fig. 2. The increase of the consumed power and discharge current indicates the increase of the power input to the plasma and the increase of the plasma density in the discharge, respectively. As shown in the figures, the consumed power and discharge current were increased not only by the increase of the input voltage, but also by the increase of the frequency of the quasi pulse power. Therefore, the increase of the SiO_x deposition rate and the improved film characteristics with increasing quasi pulse power frequency observed in Figs. 2 and 3 are related to the increased plasma density and gas dissociation caused by increasing the power consumption in the discharge. Also, increasing the input voltage of the pulse power increases the power input by increasing the power per pulse, while increasing the quasi pulse power frequency increases the power input by increasing the numbers of short pulses at a given time. Even though both of these effects can increase the plasma density, compared to the increase of the input voltage, increasing the pulse power frequency at a fixed input voltage decreases the possibility of forming a filamentary discharge by using a low input voltage, and increases the discharge efficiency by decreasing the plasma-off period (the decreased plasma-off period in the DBD decreases the loss of the accumulated charge on the dielectric surface, which is required to form an efficient DBD at a lower voltage).

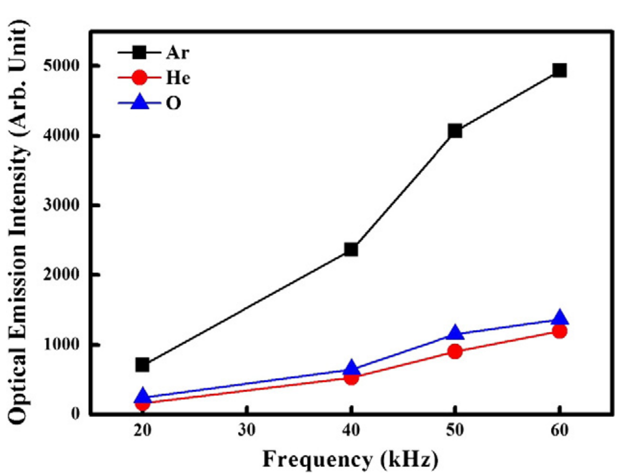

Fig. 5. OES data of He (706 nm), Ar(750.4 nm), and O(777.1 nm) measured above the substrate electrode as a function of the frequency of the pulse power, while maintaining the AC voltage of the remote-type DBD at 7 kV and the pulse voltage at 10 kV. The gas mixture is the same as that in Fig. 2.

Fig. 5 shows the OES data of He (706 nm), Ar (750.4 nm), and O (777.1 nm) measured above the substrate electrode as a function of the frequency of the quasi pulse power, while maintaining the AC voltage of the source discharge system at 7 kV pulse voltage at 10 kV [35]. As shown in the figure, increasing the frequency increased the optical emission intensities of He, Ar, and O. The optical emission

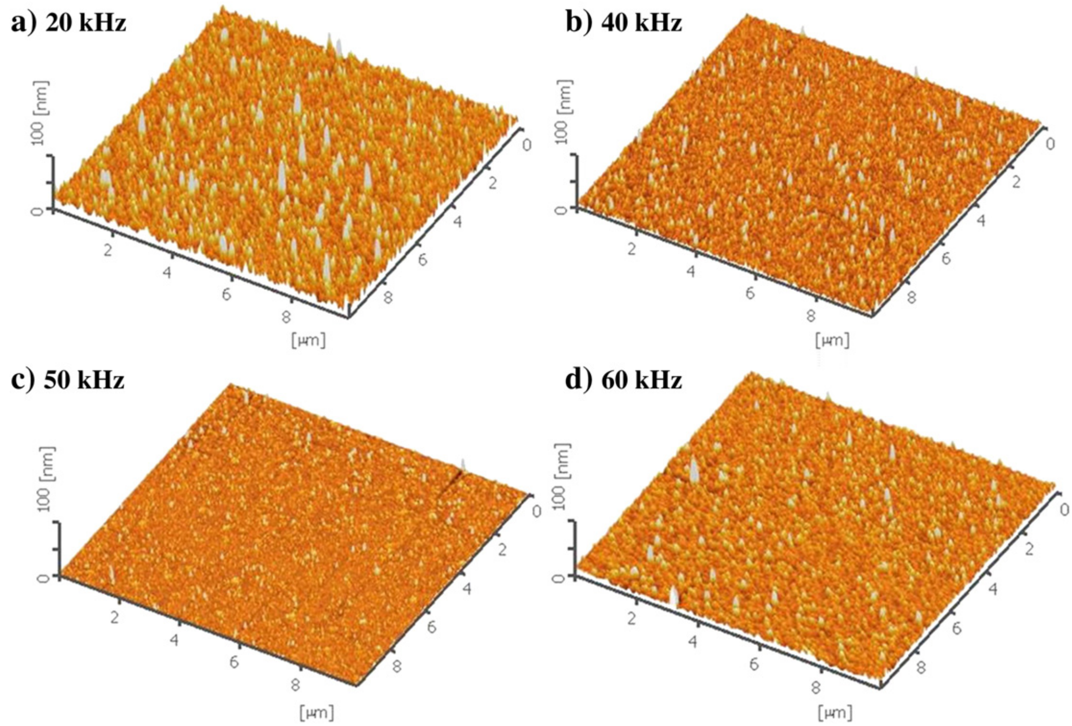

Fig. 6. Surface roughness of SiO_x film measured by AFM as a function of pulse power frequency (20–60 kHz). The input voltage of the pulse power to the direct-type DBD and AC voltage to the remote-type DBD are 10 and 7 kV, respectively. The gas mixture is the same as that in Fig. 2. The thickness of deposited SiO_x thin film was also maintained at 200 nm.

intensities of He, Ar, and O are from the gas mixed with HMDS and the variation of these intensities are related to the variation of the plasma density. As shown, the optical emission intensities increased almost linearly with increasing pulse power frequency and, therefore, the plasma density increased almost linearly with increasing frequency, as mentioned in Fig. 4.

The physical characteristics of the SiO_x deposited as a function of the pulse power frequency were further investigated by measuring the surface roughness and mechanical hardness of the deposited SiO_x. The pulse power frequency was varied, while maintaining the input voltage at 10 kV and the AC voltage of the source discharge system at 7 kV. The deposited SiO_x thickness was maintained at 200 nm. The surface roughness of the SiO_x deposited as a function of the pulse power frequency for the condition in Fig. 5 measured using AFM is shown in Fig. 6. As shown in the figure, increasing the pulse power frequency from 20 to 50 kHz decreased the surface roughness from 6.35 to 1.37 nm and further increasing the pulse power frequency to 60 kHz increased the surface roughness slightly. Fig. 7 shows the mechanical hardness measured as a function of the pulse power frequency. As shown in the figure, increasing the pulse power frequency from 20 to 60 kHz increased the mechanical hardness from 7.4 to 8.5 GPa. The improvement of the surface morphology during the thin film deposition together with the improvement of the mechanical properties afforded by the increased ion bombardment has been reported by many other researchers[26,36]. That is, during the DBD operation, even though the mean free path and the sheath thickness on the substrate are extremely small, the substrate can be bombarded by the incident ion in the plasma by the sheath potential developed on the substrate even though the bombardment energy decreased with increasing frequency. Therefore, the decrease of the surface roughness and increased mechanical hardness with increasing frequency up to 50 kHz is believed to be related to the increased bombarding ion flux applied to the substrate. (The increase of the surface roughness caused by increasing the frequency further to 60 kHz is not fully understood, but it appears to be partially related to the decreased ion bombardment energy, even with the highest bombarding ion flux to the substrate.) In addition, the improved surface roughness and increased mechanical hardness afforded by increasing the frequency is thought to be partially related to the decreased bondings, such as $Si–CH_3$ and $Si–NH_3$, in the deposited film, which make the film porous and rough (for example, the bond length of Si–O is shorter than that of $Si–CH_3$ due to the lower binding energy of $Si–(CH_3)_4$ (93.8 kcal/mol) compared to that of Si–O(129.7 kcal/mol): Si–O: 1.576–1.622 Å[37], $Si–CH_3$: 1.862 Å[38]) by increasing the dissociation and oxidation of the HMDS at the higher pulse power frequency.

Therefore, by using double discharges and increasing the frequency of the pulse power to the substrate electrode, not only the SiO_x deposition rate, but also the physical and chemical characteristics of the deposited SiO_x, could be improved, such as lower impurities, lower surface roughness, and higher mechanical hardness. These characteristics are related to the increased plasma density caused by the increased power consumption in the discharge obtained without inducing a filamentary discharge using a pulse power for the direct-type DBD for substrate biasing in addition to the source discharge system. The increased plasma density not only decreased the bondings, such as $Si–CH_3$ and $Si–NH_3$, in the deposited film, but also increased the bombardment ion flux to the substrate without decreasing the ion bombardment energy significantly.

4. Conclusions

SiO_x thin film was deposited with HMDS (400 sccm)/O_2 (20 slm)/He (5 slm)/Ar (10 slm) using a modified AP-PECVD system, which is composed of two discharge systems; a source discharge system for gas dissociation and direct-type DBD on the substrate for gas dissociation and ion bombardment. An AC voltage of 30 kHz at 5–7 kV was applied to the upper electrode (the source discharge system) and various quasi pulse voltages and frequencies were applied to the lower electrode (direct-type DBD). Increasing the lower quasi pulse voltage and frequency increased the SiO_X thin film deposition. Increasing the pulse frequency at a fixed input voltage of the direct-type DBD, while operating the source discharge system as the double discharge system, also decreased the amounts of impurities such as carbon and nitrogen, decreased the surface

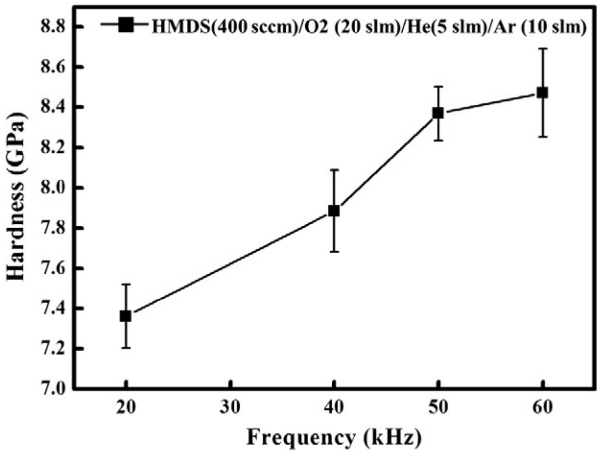

Fig. 7. Mechanical hardness measured as a function of the pulse power frequency. The thickness of deposited SiO_x thin film was also maintained at 200 nm. The quasi pulse voltage was maintained at 10 kV and AC voltage of the remote-type DBD at 7 kV.

roughness in general, and increased the mechanical hardness. The improvement of the physical and chemical properties, in addition to the increase of the SiO_x deposition rate, with increasing pulse power frequency, was related to the increased gas dissociation and ion bombardment effect caused by the increased power consumption to the discharge through the direct-type DBD. This increase in the power consumption without inducing a filamentary discharge was obtained by increasing the duty percentage by increasing the number of short pulse voltages at a given time. It is believed that by using the double discharge system, SiO_x thin film which is applicable to the thin film passivation of flexible substrates, such as polymer substrates, could be fabricated by optimizing the multilayer structure composed of organic thin film/SiO_x.

Acknowledgments

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