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Silicon nitride deposited by laser assisted plasma enhanced chemical vapor deposition for next generation organic electronic devices

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ABSTRACT

Deposition of stoichiometric silicon nitride without damage and stress at low temperature using plasma enhanced chemical vapor deposition (PECVD) is an important issue in the application to various areas such as microelectronics, micro-electromechanical system (MEMS), etc. In this study, the effects of laser assistance during PECVD (LAPECVD) of silicon nitride on the physical and chemical characteristics of deposited Si_3N_4 film were investigated. The LAPECVD assisted by 193 nm laser at 80 °C showed higher deposition rates compared to PECVD due to the enhanced dissociation of the reactant gases. In addition, the stoichiometric ratio of N/Si and the residual stress of the deposited silicon nitride film were improved. When the silicon nitride was directly deposited on the organic light emitting diode (OLED) for thin film passivation, no electrical damage was observed for LAPECVD possibly due to the coverage of a thin silicon nitride layer on the OLED surface by laser assisted deposition while conventional PECVD showed a damage of the device due to ion bombardment by direct exposure to plasma. We believe the LAPECVD system can be used for various next-generation microelectronic industries where high quality film deposition with minimized damage during PECVD at low temperature is required.

1. Introduction

Silicon nitride is one of the most widely used materials in microelectronics as dielectric layer, diffusion barriers, passivation layers, etc. and micro-electro-mechanical systems (MEMS) as structural materials due to its excellent chemical, mechanical, and optical properties [1–8]. Especially, among the various deposition methods of silicon nitride, plasma enhanced chemical vapor deposition (PECVD) is still widely used because of its high deposition rate at low process temperatures with excellent film uniformity over large substrates, and which is an essential requirement for current semiconductor and display industry [9–12]. However, conventional PECVD has disadvantages for recent applications to flat panel and flexible displays and MEMS devices. For examples, the ion bombardment during the plasma deposition process causes electrical damage to the devices, the residual stress in the film causes local peeling, delamination, cracking, or deformation of film, and low temperature deposition for flexible displays results in formation of nonstoichiometric silicon nitride film because of its low gas dissociation rate [13–18].

In this research, to overcome these problems, a novel PECVD system assisted by a 193 nm argon fluoride (ArF) laser, that is, a LAPECVD system was investigated for the deposition of silicon nitride at a low temperature using a gas mixture of $SiH_4/NH_3/N_2$. The silicon nitride thin films deposited by LAPECVD improved the deposition rate, residual stress, and thin film density (high dry etching resistance) compared to conventional PECVD under the same conditions. Finally, when the silicon nitride was deposited on organic light emitting diodes (OLED) devices as a passivation layer, no electrical damage was found for the devices passivated with silicon nitride thin film using LAPECVD, whereas, significant degradation of device performance was observed for those passivated by conventional PECVD only.

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2. Experimental section

Fig. 1 is a schematic drawing of a LAPECVD system used in the experiment. The PECVD system for the LAPECVD is a conventional capacitively coupled plasma (CCP) -type PECVD system installed with a perforated shower head as the power electrode and the substrate holder as the opposite electrode. 13.56 MHz rf power was applied to the top shower electrode while the substrate holder was grounded. For LAPECVD, a 193 nm ArF excimer laser (IPEX-740, Light Machinery) with 150 mJ pulse energy and 100 Hz repetition frequency was located outside of the chamber and was designed to irradiate a collimated laser beam with the area of 80 mm × 5 mm along the center line of the chamber through an extruded window port which is made of CaF_2 to minimize the absorption of the laser. An optical lens for the optical emission spectroscopy (OES) was installed at the opposite side of the chamber with an oblique angle to avoid direct irradiation of the laser beam. To prevent the deposition of silicon nitride on the optical windows for laser beam and OES, nitrogen (N_2) was flown at the edges of the window ports as buffer gas, and, especially, to maintain its initial transmittance of the laser beam during the process by separating the window from the process chamber, an extruded shape window port was installed.

Silicon nitride was deposited at a low temperature of at 80 °C on silicon wafers or glass wafers using a gas mixture of $SiH_4/NH_3/N_2$ as a function of SiH_4:NH_3 ratios. The total nitrogen flow rate including the nitrogen flow at the edge of the windows was 2.0 slm. The operating pressure was 1 Torr and 13.56 MHz applied to the top electrode was also varied from 400 to 1000 Watts. The power density of the ArF laser beam was adjusted to 75 mW/mm^2. The silicon nitrides were also deposited on OLED devices and they were fabricated by the deposition of organic materials through thermal evaporation with the following sequence; 50 nm of 2-TNATA, 30 nm of NPB, 30 nm of Alq_3, 0.7 nm of LiF and 100 nm of Al cathode.

The degree of gas dissociation was analyzed with OES (AvaSpec-3648, AVANTES) through the window port installed at a slightly tilted angle to avoid direct irradiation of the ArF laser beam to the optical lens. Chemical composition of the deposited silicon nitride thin films was evaluated with X-ray photoelectron spectroscopy (XPS, MultiLab 2000, Thermo VG, Mg Kα source) after calibrating peaks with C 1*s* at 285 eV. Film density was characterized by comparing the etch rates of the deposited silicon nitride films using a 13.56 MHz inductively coupled plasma (ICP) etcher with a gas mixture of CF_4 and O_2 [19–21]. The surface morphology of silicon nitride thin films was measured with an atomic force microscope (AFM, XE-100, Park System) with the non-contact measurement mode. The residual stress of the films was measured with a stress measurement system (FSM500TC, Frontier Semiconductor) by measuring the curvatures of silicon wafer (substrate) before and after silicon nitride deposition using dual wavelengths of 780 and 650 nm. Finally, to analyze ion bombardment damage, electrical characteristics of OLED devices were compared before and after the silicon nitride thin film deposition.

3. Results and discussion

Fig. 2 shows deposition rates of silicon nitride thin film deposited by PECVD and LAPECVD measured as functions of NH_3/SiH_4 gas flow ratios (Fig. 2a) and plasma rf power (Fig. 2b). For Fig. 2a, the NH_3/SiH_4 gas flow ratio was varied by changing NH_3 flow rate at the fixed SiH_4 flow rate of 300 sccm while maintaining the rf power at 1000 W and, for Fig. 2b, the rf power was varied from 400 to 1000 W while keeping the NH_3:SiH_4 ratio at 1.5:1. For the LAPECVD, a fixed ArF laser power of 75 mW/mm^2 was used. As shown in Fig. 2a, the deposition rate of silicon nitride was increased with increasing the NH_3/SiH_4 ratio at the fixed SiH_4 flow rate of 300 sccm. As shown in Fig. 2b, the deposition rate of silicon nitride was also increased with increasing the rf power. In both Fig. 2a and b, silicon nitride thin films deposited by LAPECVD were higher than those deposited by PECVD for all process conditions indicating that the assistance of the laser in plasma leads to the enhancement of reactive gas dissociation. In fact, as shown in Fig. S1 (Supplementary Information), when the dissociation of reactive gases from a NH_3/SiH_4 gas mixture was observed using OES, it showed that the addition of an ArF laser during a NH_3/SiH_4 plasma significantly enhanced the dissociation of the reactive gases. In our plasma conditions, the rf power above 1200 W and $NH_3/SiH_4 \geq 2$ showed unstable plasma dissociation, therefore, the process condition of the rf power of 1000 W with the gas ratio of $NH_3/SiH_4 = 1.5$, shown as the green box in Fig. 2a and b, was chosen as the optimized silicon nitride deposition condition. The blue line in Fig. 2b shows the optical transmittance of silicon nitride thin films deposited by the LAPECVD process. The thickness of silicon nitride was ~300 nm and the optical transmittance was measured at the

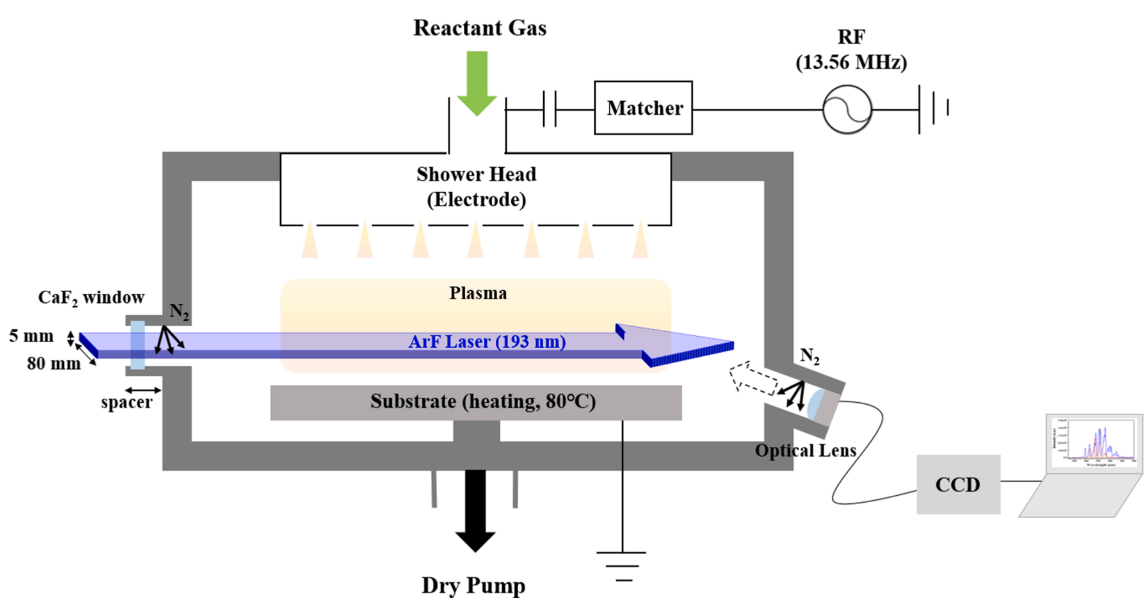

Fig. 1. Schematic drawing of the LAPECVD system used in the experiment. On the conventional PECVD system, a laser beam with the area of 80 mm × 5 mm focused using an optical system was positioned to pass along the center line of the chamber from a CaF_2 window located at the side of the chamber. To prevent the window port from being deposited by silicon nitride, an extruded window (spacer) was installed and nitrogen was continuously flown at the edge of the window.

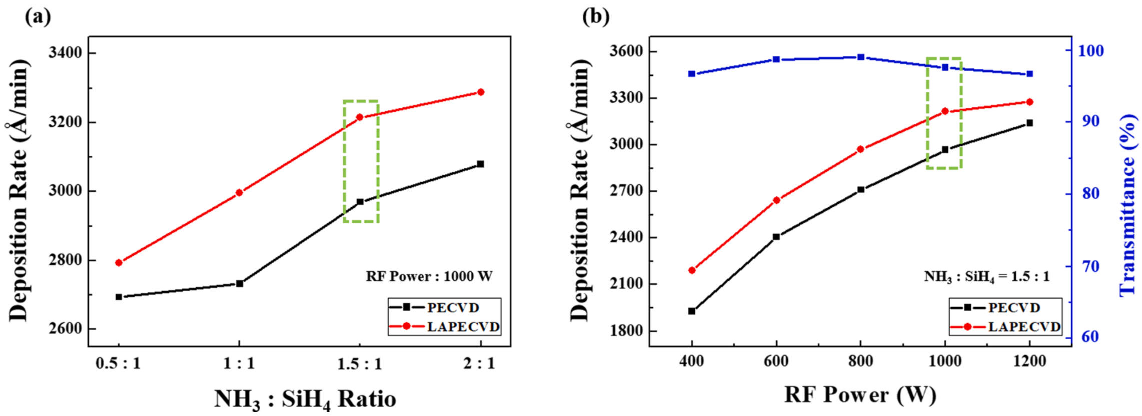

Fig. 2. Deposition rates of silicon nitride for PECVD and LAPECVD, that is, with and without a fixed ArF laser power density of 75 mW/mm^2, respectively, measured as functions of a) NH_3:SiH_4 ratio and b) plasma rf power. For a), the deposition rate of silicon nitride was investigated at a fixed SiH_4 flow rate of 300 sccm while varying the NH_3 flow rate. The blue line in b) shows the optical transmittance of silicon nitride thin films deposited by the LAPECVD process. Green dot squares in a) and b) are the optimized conditions for silicon nitride deposition. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

wavelength of 550 nm. As shown in Fig. 2b, the optical transmittances deposited by LAPECVD were higher than 96% for all conditions.

The physical and chemical properties of the silicon nitride thin films deposited by PECVD and LAPECVD were investigated. Fig. 3a shows the residual stress of the silicon nitride thin films deposited by LAPECVD with increasing rf power for NH_3:SiH_4 = 1.5:1. As shown in Fig. 3a, the residual stress of the silicon nitride thin films deposited by LAPECVD was decreased with increasing rf power reaching 147 MPa at the rf power of 1000 W from 288 MPa at 400 W. As shown in the yellow box in Fig. 3a, the residual stress of the silicon nitride thin film deposited by the PECVD at 1000 W was 222 MPa, therefore, the silicon nitride deposited by LAPECVD showed ~34% lower residual stress. To investigate the reason for the lower residual stress in the silicon nitride thin film deposited by LAPECVD compared to that deposited by PECVD, the ratio of Si:N in the deposited film was investigated using XPS. Fig. 3b shows the XPS narrow scan data of Si 2p and N 1s measured for the silicon nitride films deposited for PECVD and LAPECVD with the conditions in the yellow box of Fig. 3a. Due to the surface oxidation during the exposure of the deposited silicon nitride to the air environment, the very surface of the deposited silicon nitride thin films was sputtered using Ar^+ ion gun for 15 s before the measurement. As shown in Fig. 3b the ratio of Si:N was 54.07:45.93 for the silicon nitride deposited by LAPECVD while that for the PECVD was 61.06:38.94, therefore, even though stoichiometric silicon nitride thin film (Si_3N_4) was not deposited for both methods, higher nitrogen percentage which is closer to the stoichiometric silicon nitride could be obtained by LAPECVD possibly due to the higher reactive gas dissociation as observed in Fig. S1 in supplementary information. The lower residual stress of the silicon nitride deposited by LAPECVD compared to that by PECVD appears to be related to the improved chemical composition near to stoichiometric silicon nitride.

Additional film properties of the silicon nitrides deposited by LAPECVD and PECVD with the optimized conditions in Fig. 2 were investigated through the measurement of surface roughness and dry etch rate under CF_4/O_2 plasma. As shown in Fig. 4a, the silicon nitride thin films deposited by LAPECVD had the root-mean-square (RMS) surface roughness value similar to that of the film deposited by PECVD (1.18 and 1.16 nm for LAPECVD and PECVD, respectively). This result may indicate that the ArF laser assist during the PECVD does not generate additional particles in the plasma during the deposition. In addition, when the chemical etch resistance of the silicon nitrides deposited by LAPECVD and PECVD was investigated by etching using a CF_4/O_2 plasma, as shown in Fig. 4b, the silicon nitride thin films deposited by LAPECVD showed higher etch resistance of the average etch rate of 20 nm/min compared to that of 24 nm/min for the PECVD. The lower dry etch rate for the silicon nitride deposited by LAPECVD is related to the

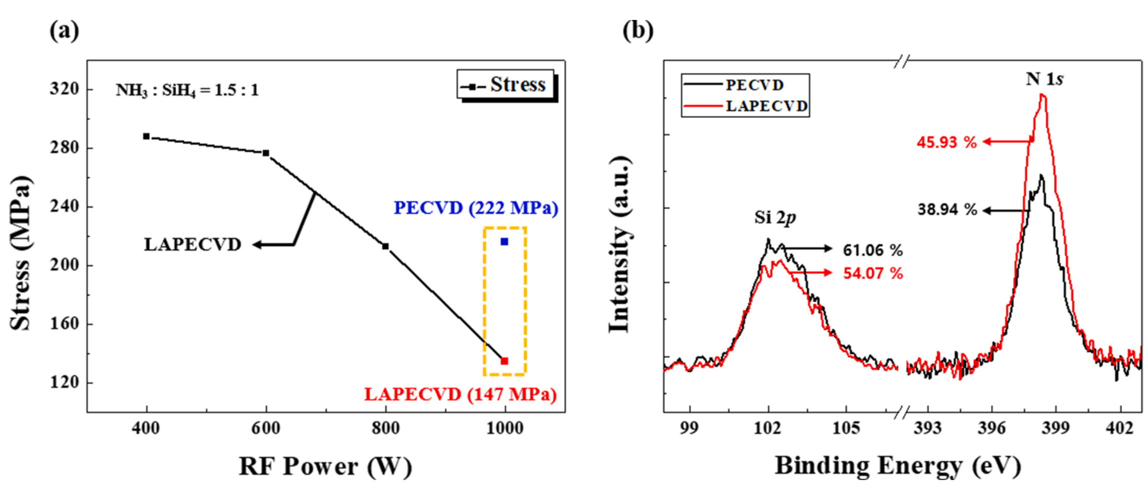

Fig. 3. (a) Residual stress of silicon nitride films with plasma rf power. (b) XPS narrow scan data of Si 2*p* (102 eV) and N 1*s* (398 eV). The numbers in b) are the atomic percentages of N (1*s*) and Si (2*p*) of the silicon nitride thin films deposited by PECVD and LAPECVD. The deposition conditions in b) are the optimized conditions in Fig. 2.

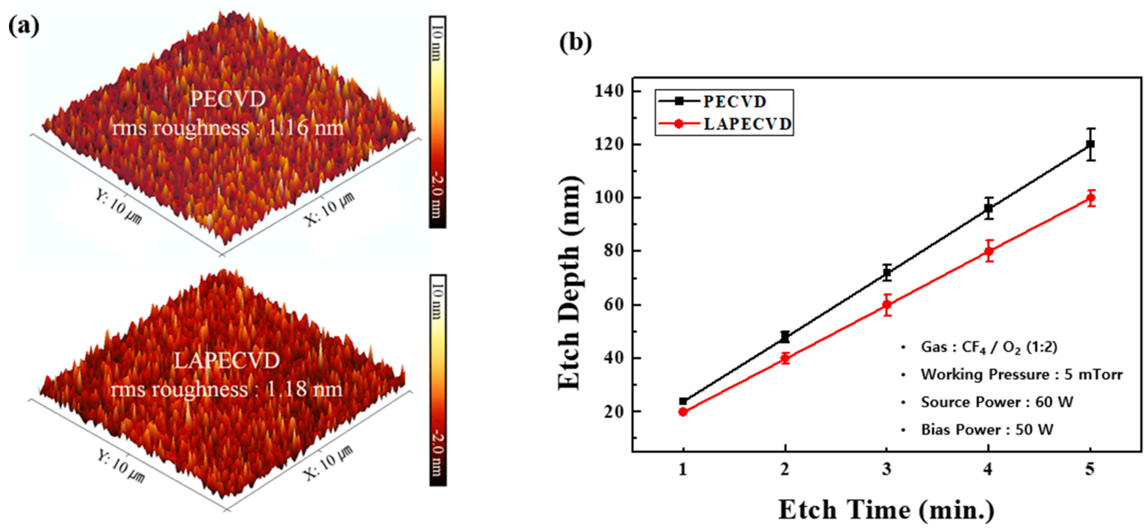

Fig. 4. a) RMS surface roughness measured by non-contact AFM with 10 × 10 μm of scan area and b) dry etch characteristics conducted using an ICP system with a gas mixture of CF_4 and O_2 (1:2) at 5 mTorr of working pressure for silicon nitrides deposited by LAPECVD and PECVD with the optimized conditions in Fig. 2.

higher film density for LAPECVD due to higher nitridation of the film [22].

For a qualitative analysis of damage caused on substrates during the deposition of silicon nitride film with the LAPECVD process, the silicon nitride thin films were deposited on OLED devices as a passivation layer, and the current efficiency of the OLED device, which is closely related with the integrity of organic layer, was measured [6]. Fig. 5a shows a schematic drawing of the OLED device structure with a bottom-emission structure used in this study. To evaluate the damage caused only by the silicon nitride deposition process and to minimize any other

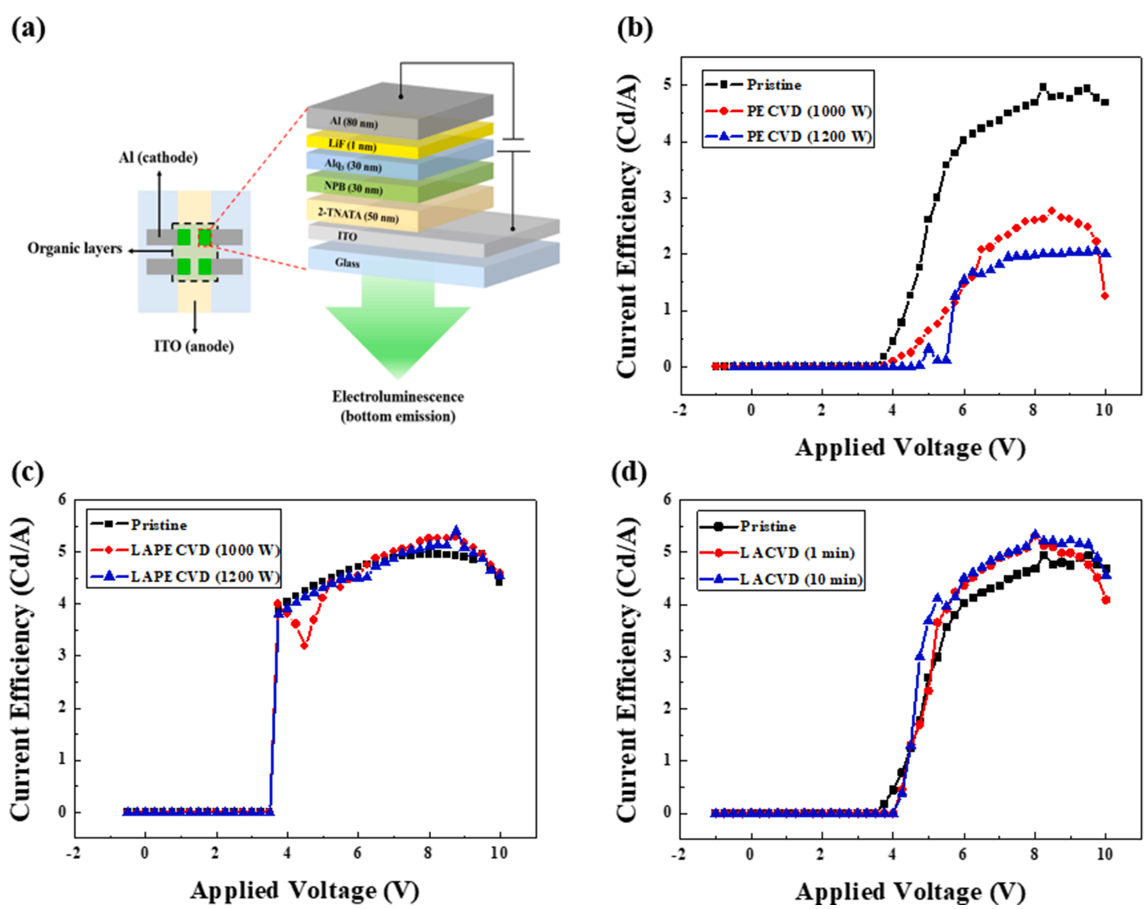

Fig. 5. Evaluation of OLED device electrical damage during the passivation process by silicon nitride deposition using LAPECVD and PECVD. a) Schematic drawing of the OLED device fabricated through thermal evaporation of organic materials on ITO glass. Characteristics of current efficiency vs. applied voltage for the OLED devices before and after deposition of silicon nitride thin films on OLED devices as a passivation layer by b) PECVD with rf powers of 1000 and 1200 W for 1 min, c) LAPECVD with rf power of 1000 and 1200 W for 1 min, and d) Laser CVD (LACVD) only for 1 and 10 min. Other deposition conditions are the same as those in Fig. 2.

environmental effect on device performance, the characteristics of current efficiency vs. applied voltage for the OLED devices were measured right after the OLED device fabrication and deposition of the silicon nitride thin films. As shown in Fig. 5b, the OLED device before the silicon nitride deposition (pristine) exhibited the maximum current efficiency of 4.95 (Cd/A) at the applied voltage of 8 V, and then, the further increase of applied bias voltage lowered the current efficiency, and which is the general characteristic of OLED devices [23–25]. However, after deposition of silicon nitride by PECVD at 1000 W of rf power for 1 min, significant degradation of maximum current efficiency over 44% (4.95–2.75 Cd/A) as well as lowered overall current efficiency was observed, and, the increase of rf power to 1200 W resulted in further degradation over 59% (4.95–2 Cd/A). In the case of OLED devices with the silicon nitrides deposited by LAPECVD, as shown in Fig. 5c, the current efficiency of OLED devices maintained its original luminance performance and little change of current efficiency vs. applied voltage was found even at 1200 W of rf power. To understand no-damage on OLED device by the LAPCVD process, a silicon nitride layer was deposited with the ArF laser only, that is, by the laser assisted CVD (LACVD), through the laser dissociation of the NH_3/SiH_4 for 1 and 10 min. As shown in Fig. 5d, the OLED devices with silicon nitride deposited by LACVD did not show any degradation of the device properties even after 10 min deposition (250 nm-thick silicon nitride was deposited by 10 min LACVD). It is believed that the no-damage observed on the OLED device with the silicon nitride deposited by LAPECVD is also related to the effect of initial OLED device surface coverage by silicon nitride with LACVD during LAPECVD (even though the laser and the plasma were turned-on almost at the same time during the LAPECVD, the ArF laser was turned-on a little earlier within 3 s before the plasma was turned-on). By protecting the OLED device surface with a thin silicon nitride by LACVD, the damage by the ion bombardment from the plasma during the LAPECVD could be eliminated. Additionally, to clearly understand the effect of a protecting silicon nitride layer by LACVD before PECVD on the damage of OLED device during the LAPECVD process, silicon nitride layers was sequentially deposited on OLED device by 1st LACVD ~20 nm thick and by 2nd PECVD using 1000–1200 W, and the results showed no damage on OLED device for 1000 W while some damage is showing for 1200 W indicating the importance of a protecting buffer layer by LACVD before the plasma exposure (Fig. S2, Supplementary Information).

4. Conclusion

The effects of ArF laser assistance during the conventional PECVD using NH_3/SiH_4, that is, LAPECVD, on the characteristics of deposited silicon nitride thin film were investigated. Comparing the conventional PECVD, the LAPECVD showed higher silicon nitride deposition rate, more stoichiometric silicon nitride thin film with higher nitridation, lower residual film stress, and higher film density estimated by the dry etch rate using CF_4/O_2 plasma. These improved characteristics of the deposited silicon nitride thin films by LAPECVD were related to the enhanced dissociation of NH_3/SiH_4, therefore, by increasing reactive nitrogen and silicon based radicals in the plasmas. Especially, when the silicon nitride layer was deposited directly on OLED devices as a thin film passivation layer, the devices with a silicon nitride layer deposited by LAPECVD did not show any electrical damage while those with a silicon nitride layer by PECVD showed an electrical damage due to the ion bombardment from the plasma. No damage on OLED device with the silicon nitride layer by LAPECVD was related to a thin silicon nitride deposition as a protection layer before the plasma exposure by LACVD on OLED devices during the LAPECVD process. It is believed that the thin film deposition technology using LAPECVD can be useful for depositing various thin films requiring enhanced materials properties including less surface damage on substrates such as a passivation layer for the next generation flexible organic electronic devices, etc. However, due to the laser absorption along the laser path, in order to apply the LAPECVD to large area plasma systems, a method for solving the uniformity issue with laser irradiation distance must be considered (Fig. S3, Supplementary Information).

CRediT authorship contribution statement

Ki Hyun Kim: Conceptualization, Investigation, Writing - original draft. **Ki Seok Kim:** Conceptualization, Methodology, Investigation, Writing - review & editing. **You Jin Ji:** Investigation. **Ji Eun Kang:** Investigation. **Geun Young Yeom:** Supervision, Project administration, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.apsusc.2020.148313.

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