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Nitriding process for next-generation semiconductor devices by VHF (162 MHz) multi-tile push-pull plasma source

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ABSTRACT

For the low power and high-performance semiconductor devices, a silicon oxynitride (SiO_xN_y) layer is required as the gate sidewall spacer material by replacing oxygen of the silicon oxide (SiO_2) sidewall layer with nitrogen through a plasma nitriding process. In this study, as a plasma nitriding process, instead of conventional radio frequency plasma nitriding utilizing high frequency (HF; 13.56 MHz, etc.) plasmas, a very high frequency (VHF) plasma operated at 162 MHz with a multi-tile push-pull plasma source has been used in nitriding the SiO_2 layer at room temperature and the effects of the VHF (162 MHz) plasma on SiO_xN_y formation from a SiO_2 layer and electrical characteristics of the SiO_xN_y formed by the plasma nitridation were investigated. The use of the VHF (162 MHz) multi-tile push-pull plasma formed ~10 nm thick SiO_xN_y with a very high nitrogen percentage of ~24% on the SiO_2 layer surface. Also, when the surface roughness of the SiO_xN_y and electrical characteristic of MOS capacitors fabricated with the SiO_xN_y formed by the VHF (162 MHz) plasma were compared with those formed by a capacitively coupled plasma at 60 MHz, a lower surface roughness and much lower leakage current of MOS capacitor could be obtained.

1. Introduction

Nitridation has been widely applied to various semiconductor devices for gate dielectrics, surface/interface treatment, etc. [1–4]. Especially, in silicon semiconductor device processes, nitridation has been used to form a SiO_xN_y gate dielectric layer [5–10]. A SiO_xN_y layer has been applied to prevent the diffusion of p-type dopant such as boron through a thin gate oxide film [11–16] and to reduce the severe sidewall oxide spacer etching during the self-aligned gate formation as the size of semiconductor devices is continuously scaled down. In order to meet the process requirements of the SiO_xN_y layer, various stuides in forming a high quality nitrogen containing SiO_xN_y layer (with a high nitrogen and a low hydrogen content) have been reported using thermal nitridation using NO, N_2O, and NH_3 [5–6,16–18], and plasma nitridation using conventional high frequency (HF, 13.56 MHz) plasmas [7–10,19–20]. However, thermal nitriding processes can degrade the performance of a semiconductor device due to the use of high temperatures (600–1500 °C), and plasma nitriding processes can degrade the semiconductor material due to the ion bombardment and hydrogen inclusion. For the plasma nitridation with N_2 to remove possible damage by hydrogen, it is known to be difficult to form a high nitrogen containing SiO_xN_y layer because of insufficient decomposition of N_2 due to high electron-impact dissociation energy (24.3 eV) of nitrogen molecules [21].

Among the various plasma parameters, excitation frequency is one of the parameters that can change plasma properties significantly. During the deposition of silicon-based layers, it has been found that, the use of higher excitation frequency (that is, the use of a very high frequency (VHF) plasma instead of a HF plasma) is more effective for enhancing the film properties with high deposition rates due to improved plasma properties (e.g., high electron density, high radical generation rate, less ion bombardment, etc.) [22–28]. Therefore, for plasma nitridation, it would be also helpful to generate a plasma with VHF instaed of HF to sufficiently decompose N_2 molecule without ion bombardment at a low temperature. However, the standing wave effect at the higher frequency operation can be a critical limitation for plasma

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uniformity over a large area. In addition, the operation of a plasma source at a higher excitation frequency increases the inductive impedance of the plasma circuit and makes it difficult to match the plasma system impedance during the plasma operation consistently [29].

In previous researches, to compensate the plasma non-uniformity and to remove the match-related problem during the operation of VHF plasmas, a multi-tile push-pull plasma source composed of segmented electrode sets (floating push/pull power electode set) has been investigated and, using the VHF (162 MHz) multi-tile push-pull plasma, a uniform and stable high density plasma could be obtained [30–32]. In addition, for a thin film passivation layer for organic light emitting diodes, high quality silicon nitride (Si_3N_4) thin films could be deposited without damaging the organic devices by minimizing ion bombardment at high deposition rate through low electron temperature (T_e), high vibrational temperature (T_v), and high ion density (n_i) using a VHF (162 MHz) N_2 plasma [30]. In this study, the effect of a N_2 plasma generated by the VHF (162 MHz) multi-tile push-pull plasma source on the nitridation of SiO_2 for the formation of a high quality SiO_xN_y layer has been investigated. It has been found that, by using the VHF (162 MHz) multi-tile push-pull plasma, a SiO_xN_y layer with a very high nitrogen percentage (24.5%) and a low surface roughness could be deposited at room temperature. In addition, when the leakage current of a metal oxide semiconductor (MOS) capacitor fabricated with a SiO_xN_y layer using the VHF (162 MHz) plasma source was compared with that fabricated by a capacitively coupled plasma (CCP) operated at 60 MHz, a much lower leakage current was observed for the MOS device by the VHF plasma.

2. Experimental section

2.1. VHF (162 MHz) multi-tile push-pull plasma source and nitriding process conditions.

The VHF (162 MHz) multi-tile push-pull plasma source used for plasma nitriding processes is shown in Fig. 1. The multi-tile push-pull plasma source was composed of 4 pairs of electrodes, and where, one electrode pair is consisted of two adjacent same size rectangular shaped power electrodes (push/pull electrodes) connected to the power splitter. Each push/pull electrode pair is powered equally by a power splitter connected to the VHF (162 MHz) power generator and, during each VHF voltage cycle, high/low voltages are supplied alternatively to two push/pull electrodes to generate plasmas under the plasma source. The details of the VHF multi-tile push-pull plasma source can be found elsewhere [32–33].

For the plasma nitriding process, the 50 nm-thick SiO_2 layer deposited at 150 °C by plasma enhanced chemical vapor deposition on silicon wafer was exposed at room temperature to the VHF power varied from 420 to 2520 mW/cm^2 and the operating pressure of N_2 varied from 5 to 25 mTorr for 2 min to form SiO_xN_y layers. To compare the properties of the SiO_xN_y layer formed by the VHF (162 MHz) multi-tile push-pull plasma with those formed by a CCP operated at 60 MHz, the SiO_2 layers were exposed to the same process conditions (2100 mW/cm^2 of rf power and 20 mTorr of operating pressure for 2 min) at room temperature.

2.2. Device fabrication

To fabricate a MOS capacitor, SiO_xN_y layers was formed on the 50 nm–thick SiO_2 by the nitriding process described above with the VHF (162 MHz) multi-tile push-pull plasma source and the conventional CCP source operated at 60 MHz. After the formation of SiO_xN_y, a portion of the SiO_xN_y/SiO_2 layer was removed using a HF/deionized water solution (1/200) to expose Si, and an aluminum (50 nm) layer was deposited on the SiO_xN_y and on the exposed Si wafer surface (Fig. S1, Supplementary Information) to form a MOS capacitor. In order to clearly compare the elcetrical properties of MOS capacitors fabricated using the VHF (162 MHz) multi-tile push-pull plasma and the conventional CCP operated at 60 MHz, the MOS capacitors were fabricated to a size of 1 cm × 1 cm.

2.3. Characterization

To investigate the plasma characteristics, the ion density was measured using a Langmuir probe (ALP-150, Impedans) at the location

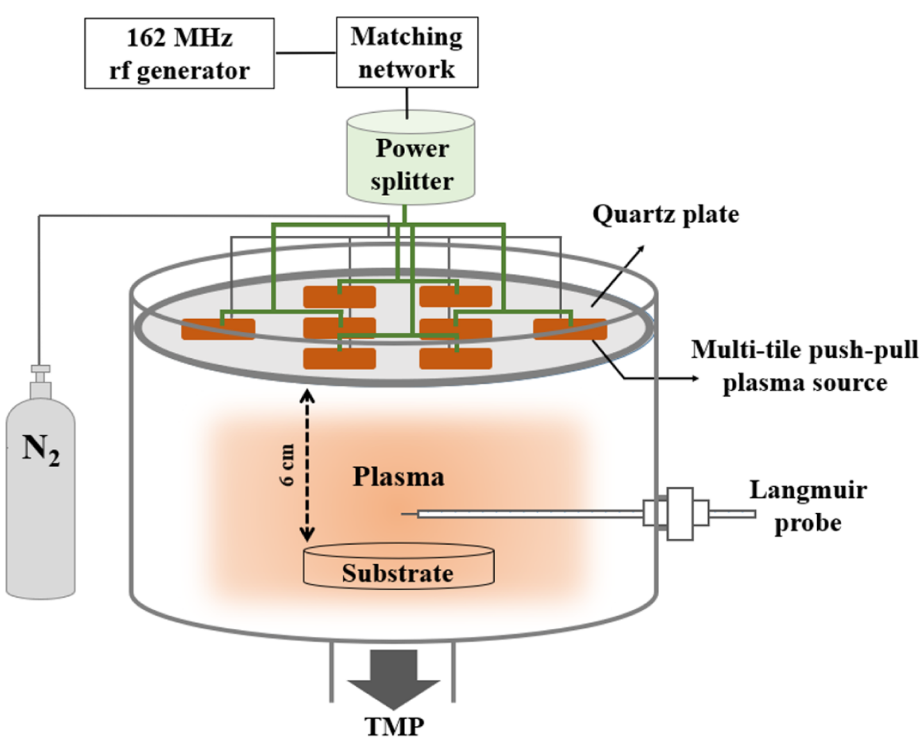

Fig. 1. Schematic drawing of the plasma nitriding system using a VHF (162 MHz) multi-tile push-pull plasma source.

between the plasma source and the substrate and at the center of the substrate where the nitriding process proceeds. Plasma density along the chamber centerline was estimated by measuring the ion saturation current using a homemade electrostatic probe biased at −30 V (Fig. S6, Supplementary Information). The ion saturation current was obtained using a Keithley 2000 after removing the rf noise through an rf filter. The atomic compositions of SiO_xN_y layers were measured by XPS (MultiLab 2000, Thermo VG, Mg Kα source) after the peak energies were calibrated by selecting the C 1s peak at 284.5 eV and the depth profiling analysis was conducted with a small area (SA)-XPS mode within the region by sputter etching the 3 mm × 3 mm area for 30 s (1 cycle) intervals using an in-situ Ar^+-ion gun. The profiling depth per cycle was ~2.62 nm/cycle (Fig. S2, Supplementary Information). The RMS surface roughness of SiO_xN_y was characterized by atomic force microscopy (AFM, Dimension 3100, Veeco). To investigate surface morphology and element distribution of the SiO_xN_y layer, EF-TEM (JEM-2100F, JEOL) mapping with a field emission gun operated at 200 keV was conducted. The leakage current of MOS capacitors were measured using a semiconductor parameter analyzer (Agilent 4155C) in a probe station (MST-5000).

3. Results and discussion

Fig. 2a and b show the ion density of N_2 plasma at different rf powers (420–2100 mW/cm^2 at 20 mTorr) and different operating pressures (5–25 mTorr at 2100 mW/cm^2), respectively, using the 162 MHz multi-tile push-pull plasma source. As shown in Fig. 2a, as the rf power was increased from 420 to 2100 mTorr at an operating N_2 pressure of 20 mTorr, the ion density was continuously increased and a high-density plasma (> 10^{11} cm^{-3}) was obtained at 2100 mW/cm^2 (at the rf power above 2100 mW/cm^2, the plasma was unstable and the ion density could no longer be increased). As shown in Fig. 2b, when the operating pressure of N_2 was increased from 5 to 20 mTorr at the rf power of 2100 mW/cm^2, the ion density was also gradually increased even though the ion density was slightly decreased at 25 mTorr. From the above results, as an optimized plasma nitriding process condition, the condition of 2100 mW/cm^2 of rf power and 20 mTorr of operating pressure was selected and this trend was also consistent with the following results on XPS composition analysis of the SiO_xN_y layers deposited with various the nitriding conditions in Fig. 2.

Fig. 3 shows the atomic percentages of Si, O, and N of SiO_xN_y layers obtained by the XPS analysis after plasma nitriding on the SiO_2 substrate by the VHF (162 MHz) multi-tile push-pull plasma using the process conditions in Fig. 2. The nitriding was continued for 2 min at room temperature. Fig. 3a shows the atomic percentages of Si, O, and N with increasing rf power at 20 mTorr of operating pressure. For the pristine SiO_2 substrate without the nitriding process, the atomic percentages of Si and O were 33.4 and 66.6%, respectively. As the rf power was increased from 420 to 2100 mW/cm^2, the atomic percentage of N was continuously increased from 7.4 to 24.5%. During the increase of rf power from 420 to 2100 mW/cm^2, the atomic percentage of Si was maintained consistently despite the continuous increase of N and the atomic percentage of O was continuously decreased from 66.6 to 49.0%, indicating that the nitrogen atoms were substituted for oxygen atoms and changing from SiO_2 to SiO_xN_y. In addition, when the rf power was increased further from 2100 to 2520 mW/cm^2, the atomic percentage of N remained similar at 23.4%, indicating that the nitridation rate was saturated at the rf power of ~2100 mW/cm^2. Fig. 3b shows the atomic percentages of Si, O, and N with increasing pressure at 2100 mW/cm^2 of rf power. As the operating pressure was increased from 5 to 20 mTorr, the atomic percentage of N increased from 15.3 to 24.5%. Further, when the operating pressure was increased to 25 mTorr, no further increase in the N content was observed on the SiO_xN_y layer. Therefore, the process conditions in the plasma nitriding process by the VHF (162 MHz) multi-tile push–pull plasma were optimized to 2100 mW/cm^2 of rf power, 20 mTorr of operating pressure, and 2 min of operating time at room temperature. The reason of the operating time optimized to 2 min was also related to the saturation of the atomic percentage of N after the operating time longer than 2 min (Fig. S3, Supplementary Information). Also, when the plasma nitriding process was performed using a conventional CCP operated at 60 MHz under the same process conditions, the atomic percentages of Si, O, and N of the SiO_xN_y layer formed on SiO_2 substrate were 33.9, 57.9, and 8.2%, respectively. Therefore, it was confirmed that the atomic percentage of N was much higher at VHF(162 MHz) (Fig. S4, Supplementary Information).

Fig. 4a and b show the N 1s XPS narrow scan data of the SiO_xN_y layer measured before and after the depth profiling with an in-situ Ar^+-ion gun for 1 cycle (30 s) to 3 cycles (90 s) for the SiO_xN_y layers obtained after the nitriding process at different rf powers of 1260 and 2100 mW/cm^2, respectively. The nitridation was processed under the same process conditions (20 mTorr of operating pressure, 2 min of operating time at room temperature) using the VHF (162 MHz) multi-tile push-pull plasma on a SiO_2 substrate. As shown in Fig. 4a, the peak intensity of N 1s XPS narrow scan data at 397 eV was gradually decreased with increasing depth profiling cycle of the SiO_xN_y layer formed with 1260 mW/cm^2 of rf power. On the contrary, as shown in Fig. 4b, for the SiO_xN_y layer formed with 2100 mW/cm^2 of rf power, the peak intensity of N 1s was decreased slightly after 1 cycle depth profiling (~2.62 nm), but it remained similar even after the depth profiling up to

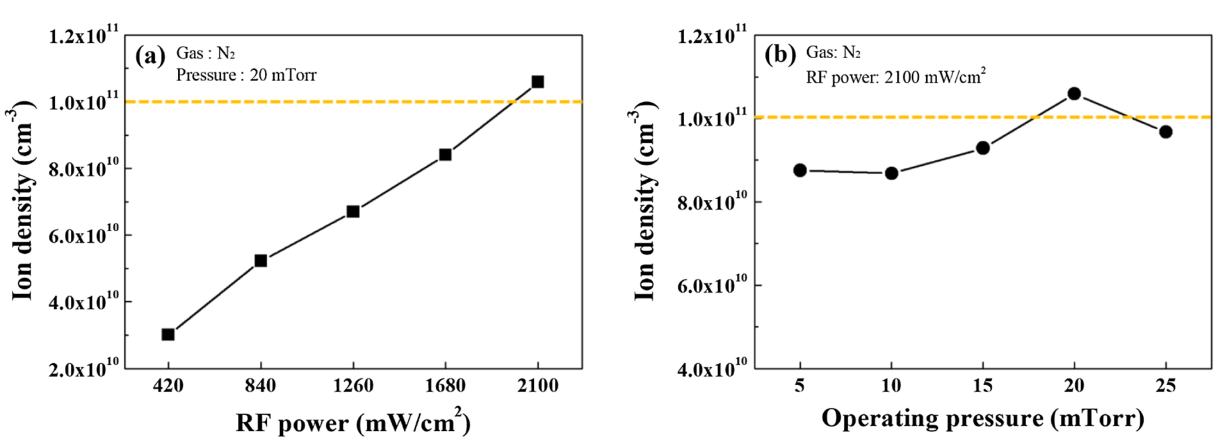

Fig. 2. Ion density of N_2 plasma generated using the VHF multi-tile push-pull plasma source as a function of (a) rf power, and (b) operating pressure measured by a Langmuir probe.

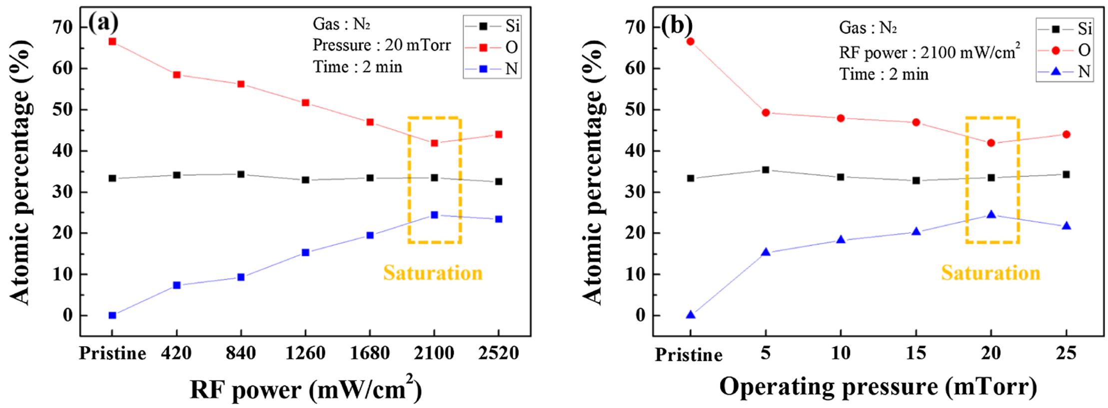

Fig. 3. Atomic percentages of Si, O, and N of SiO_xN_y layers after the nitriding process on the SiO_2 substrate as a functions of different (a) rf power, and (b) operating pressure by the VHF (162 MHz) multi-tile push-pull plasma. Atomic percentage of N was saturated at 2100 mW/cm^2 and 20 mTorr.

3 cycles (~7.85 nm) (Fig. S2, Supplementary Information). In addition to the main N 1s peak at 397 eV, there is a small bump around a 401.5 eV attributed to the N-O bonding, suggesting the formation SiO_xN_y film due to the nitridation process [34–35]. Moreover, Fig. 4b shows a higher peak intensity at 401.5 eV than peak intensity of Fig. 4a, which corresponds to the high N percentage of the SiO_xN_y layer. Fig. 4c shows the atomic percentages of N of the SiO_xN_y layer measured as the depth profiling progresses in XPS with the conditions of (a) and (b). At 1260 mW/cm^2 of rf power, the atomic percentage of N of the SiO_xN_y layer was decreased gradually from 15.4 (SiO_xN_y as-is) to 10.1% (after 3 cycles), while, at 2100 mW/cm^2 of rf power, the atomic percentage of N was decreased from 24.5 (SiO_xN_y as-is) to 20.6% after 1 cycle of depth profiling, and then it remained similar up to 3 cycles of depth profiling. Therefore, through the plasma nitriding process using the VHF (162 MHz) multi-tile push-pull plasma, a high nitrogen-containing and uniform SiO_xN_y layer could be obtained at room temperature.

Fig. 5 shows Fig. 5a the cross-sectional TEM image and Fig. 5b–d the EF-TEM element mapping data of the SiO_xN_y layer formed on top of the SiO_2 substrate by the nitriding process with the optimized process conditions (2100 mW/cm^2 of rf power, 20 mTorr of operating pressure) for 2 min at room temperature using the VHF (162 MHz) multi-tile push-pull plasma. As shown in the cross-sectional TEM image of Fig. 5a, after the formation of a SiO_xN_y layer on top of the SiO_2 substrate, no structural change except for the slight change in the brightness could be observed on the SiO_2 surface because both SiO_xN_y and SiO_2 are amorphous materials. However, when EF-TEM element mapping analysis of Si, O, and N was performed at the same position, as in Fig. 5b-d, nitrogen atoms uniformly substituted on the SiO_2 substrate to form a SiO_xN_y layer with the thickness of about ~10 nm could be observed. In addition, when the EF-TEM mapping was performed on the patterned SiO_2 for the same process conditions with the VHF (162 MHz) multi-tile push-pull plasma, excellent coverage of a SiO_xN_y layer on the sidewall of SiO_2 could be also observed even though the thickness of the SiO_xN_y layer on the sidewall of SiO_2 was somewhat lower than that of the top of SiO_2. (Fig. S5, Supplementary Information).

Fig. 6 show the AFM topograpic images before and after the nitriding process on the SiO_2 substrates by the VHF (162 MHz) multi-tile push-pull plasma and a conventional CCP operated at 60 MHz under the same process conditions (2100 mW/cm^2 of rf power and 20 mTorr of operating pressure) for 2 min at room temperature. Fig. 6a and c show that the RMS roughness of the pristine SiO_2 substrates showing 0.35 and 0.36 nm, respectively. Fig. 6b and d show the RMS roughness after the nitriding process with the VHF (162 MHz) multi-tile push–pull plasma and the conventional CCP operated at 60 MHz, and which are reduced to 0.23 and 0.31 nm, respectively. Therefore, significant decrease of RMS roughness was observed on the SiO_2 surface after the plasma nitriding process with the VHF (162 MHz) multi-tile push-pull plasma compared to that with the conventional CCP operated at 60 MHz. During the operation of plasmas, the SiO_xN_y film surface is continuously bombarded by nitrogen positive ions (N_2^+ and N^+) and, due to the ion bombardment, the surface roughness can be also decreased by compacting effect. For a given power density, the ion density at 162 MHz is about 50% higher than that at 60 MHz (Fig. S6, Supplementary Information). Therefore, lower RMS surface roughness of SiO_xN_y observed with 162 MHz multi-tile push–pull plasma compared to that with 60 MHz plasma is probably related to the higher ion density with 162 MHz multi-tile push-pull plasma.

In order to observe the electrical characteristics of the SiO_xN_y layer

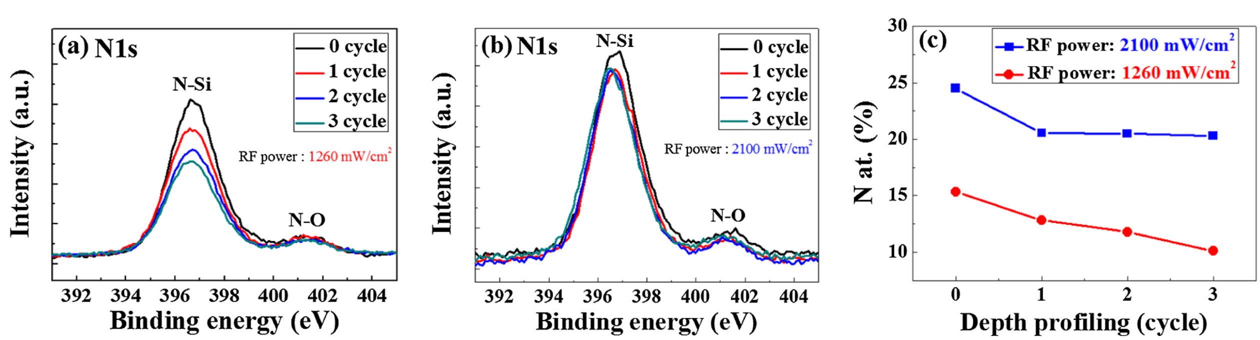

Fig. 4. XPS depth profiling analysis of SiO_xN_y after the nitriding process on the SiO_2 substrate using the VHF (162 MHz) multi-tile push-pull plasma. N 1s XPS spectra at 397 eV of SiO_xN_y formed for different rf powers of (a) 1260 mW/cm^2 and (b) 2100 mW/cm^2. The plasma was operated at 20 mTorr for 2 min. 1 cycle corresponds to profiling depth of ~2.62 nm. (c) Atomic percentage of N in the SiO_xN_y at different rf powers (1260 and 2100 mW/cm^2).

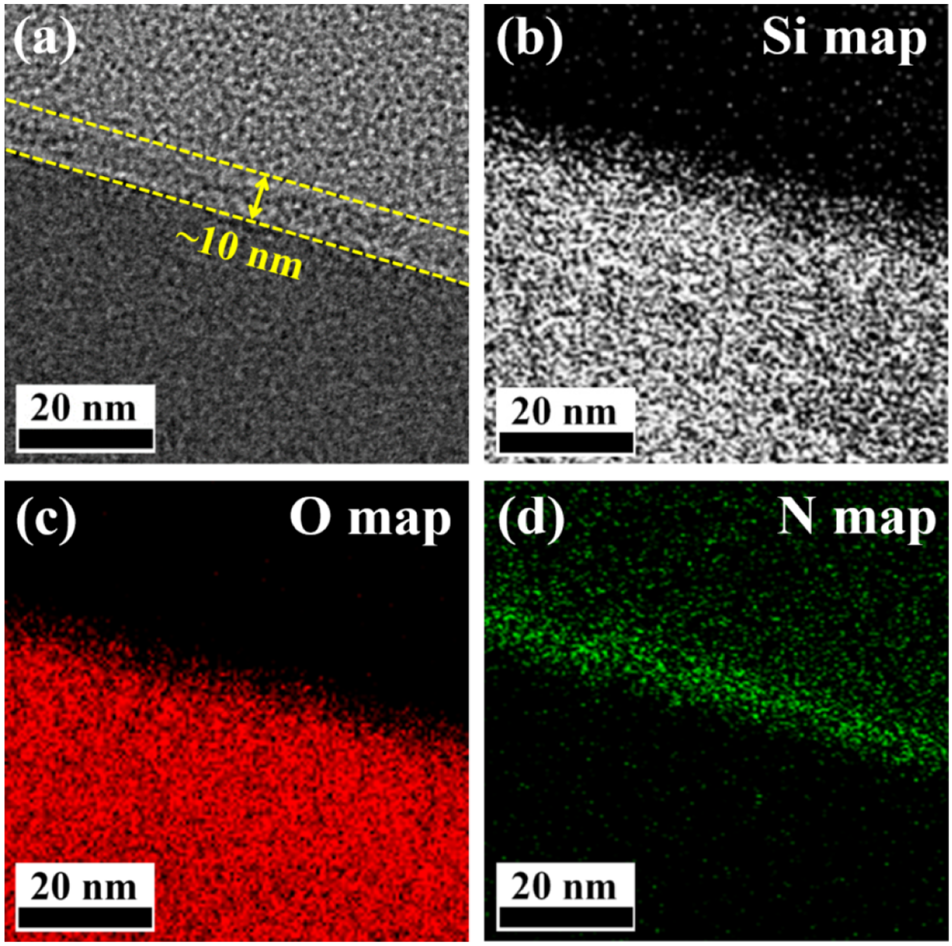

Fig. 5. (a) Cross-sectional TEM images of the SiO_xN_y layer after the nitriding process on the SiO_2 substrate by the VHF (162 MHz) multi-tile push-pull plasma and EF-TEM element mapping for (b) Si, (c) O, and (d) N.

formed after the plasma nitriding process by the VHF (162 MHz) multi-tile push-pull plasma and the conventional CCP oeprated at 60 MHz, MOS capacitors were fabricated on the 50 nm–thick SiO_2/Si substrate after the nitriding process with the same process conditions in Fig. 6. The area of the MOS capacitors was 1 cm × 1 cm. The fabrication sequency of the MOS capacitors can be seen in (Supplementary Information, Fig. S1). The leakage current curves for three different MOS capacitors fabricated on 50 nm SiO_2 (without nitridation), on the SiO_xN_y formed with 50 nm SiO_2 using the VHF plasma, and on the SiO_xN_y formed with 50 nm SiO_2 using the CCP at 60 MHz are shown in Fig. 7. The leakage current of the MOS capacitor without nitriding process on the 50 nm SiO_2 was 8.19×10^{-7} A/cm^2 at −1 V. On the contrary, after the plasma nitriding process on the SiO_2 using the CCP operated at 60 MHz, the MOS capacitor exhibited a relatively lower leakage current of 4.85×10^{-7} A/cm^2 at −1 V, and with the nitriding process on the SiO_2 using the VHF (162 MHz) plasma, the MOS capacitor exhibited the lowest leakage current of 9.55×10^{-8} A/cm^2 at −1 V. The lowest leakage current observed for the MOS capacitor fabricated with the VHF (162 MHz) plasma is believed to be related not only to the highest nitrogen-containing SiO_xN_y layer with the lowest surface roughness but also to the low possible electrical damage to the SiO_xN_y layer. Therefore, the plasma nitriding process by the VHF (162 MHz) multi-tile push-pull plasma can be an effective method in forming a high quality SiO_xN_y layer for next generation semiconductor devices.

4. Conclusions

Nitriding process is one of the important processes for next generation semiconductor device manufacturing. In this study, a plasma nitriding process has been performed on SiO_2 substrates in a high-density plasma of $> 10^{11}$ cm^{-3} by the VHF (162 MHz) multi-tile push-pull plasma source and the effect of plasma parameters on the quality of SiO_xN_y layer formed on the SiO_2 substrate was investigated and compared with that formed by a conventional CCP source operated at 60 MHz. The nitrogen content in the SiO_xN_y formed by plasma nitriding of SiO_2 substrates by the VHF (162 MHz) multi-tile push-pull plasma was saturated at an optimized conditions of 2100 mW/cm^2 of rf power, 20 mTorr of operating pressure, and 2 min of operating time. At the optimized conditions, 10 nm-thick SiO_xN_y layer with a higher atomic percentage of N (24.51%), a low RMS surface roughness of 0.21 nm, and a negligible electrical damage could be obtained. Therefore, it is found that the plasma nitriding process by the VHF (162 MHz) multi-tile push-pull plasma can be an effective method in forming a high quality SiO_xN_y layer for various next generation semiconductor devices. In addition, the characteristics of the VHF multi-tile push–pull plasma such as high nitrogen dissociation and extremely low damage to the substrate can be also essential for depositing a nitride layer using plasma-enhanced chemical vapor deposition (PE-CVD) and plasma-enhanced atomic layer deposition (PE-ALD) processes as well as a nitriding process.

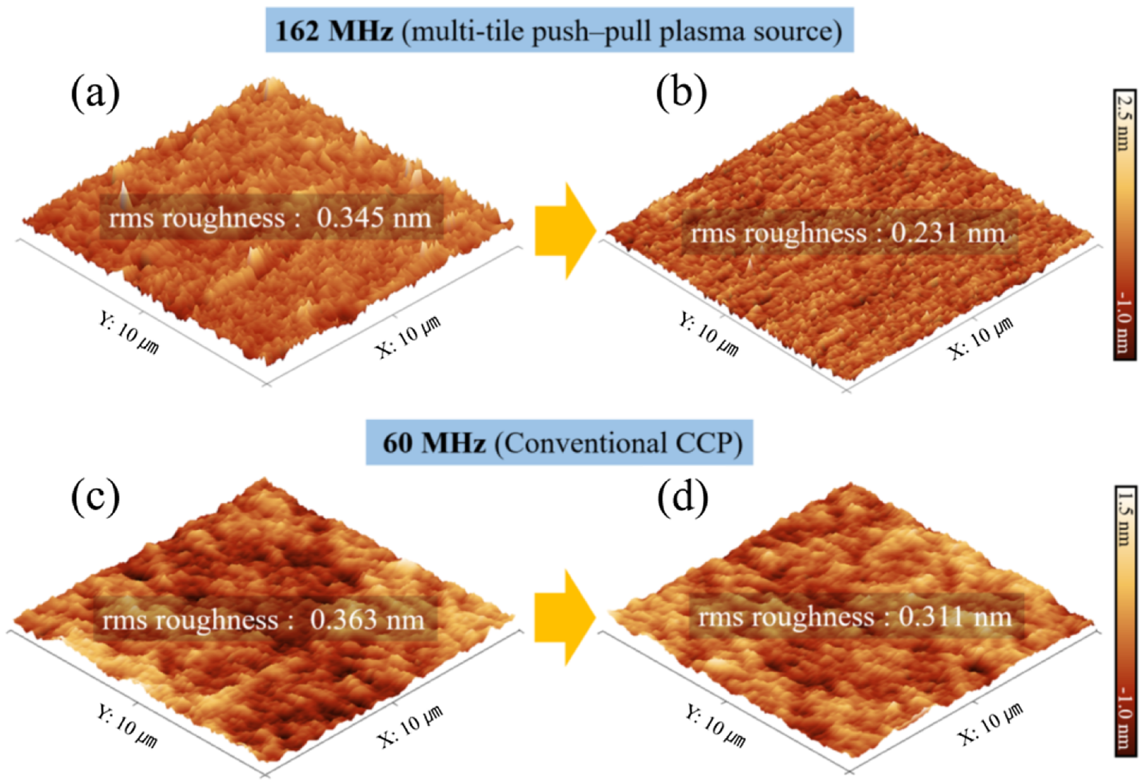

Fig. 6. Comparison of RMS roughness of SiO_xN_y formed by the VHF (162 MHz) multi-tile push-pull plasma and by a conventional CCP operated at 60 MHz under the same process conditions (2100 mW/cm^2 of rf power, and 20 mTorr of operating pressure) for 2 min at room temperature.

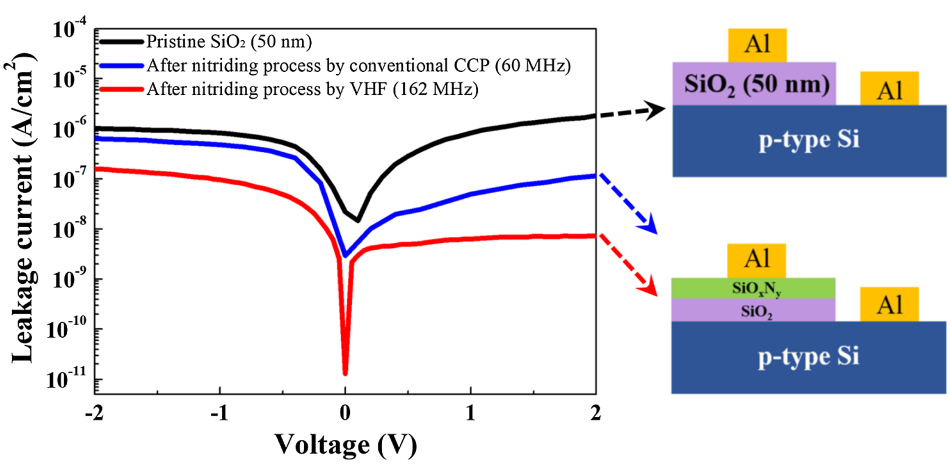

Fig. 7. Leakage currents of MOS capacitors fabricated without nitriding process on the SiO_2 substrate (black), with the plasma nitriding process on the SiO_2 substrate by the VHF (162 MHz) multi-tile push-pull plasma (red), and with the conventional CCP operated at 60 MHz (blue) under the same process conditions (2100 mW/cm^2 of rf power, 20 mTorr of operating pressure) for 2 min at room temperature. Schematics showing of MOS capacitors: without nitriding process on the SiO_2 and with nitriding process on the SiO_2. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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Appendix A. Supplementary material

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