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Effect of RF power of post-deposition oxygen treatment on HfO_2 gate dielectrics

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ABSTRACT

The radio frequency (RF) power effect of post-deposition O_2 plasma treatment on the physical, electrical, and reliability characteristics of high-*k* HfO_2 dielectric films was comprehensively investigated in this study. The experimental results indicated that increasing the RF power of post-deposition O_2 plasma treatment resulted in a stoichiometric HfO_2 film, but led to a thinner interfacial layer and the formation of new Hf–Si bonds. Additionally, the electrical performance and reliability of HfO_2 dielectric films were significantly impacted by the RF power of the post-deposition O_2 plasma treatment. As the RF power is less than 30 W, the leakage current density and time-to-breakdown of the O_2 plasma-treated HfO_2 films were improved in comparison with those of the as-deposited samples. However, further increasing RF power to exceed 50 W would cause the continuous degradation in the electrical performance and reliability due to the plasma damage induced by oxygen active spices in a plasma environment. Therefore, performing a post-deposition O_2 plasma treatment process on the as-deposited HfO_2 dielectric films can effectively improve the dielectric's properties. However, the applied RF power is an essential controlling parameter, avoiding serious plasma damage occurrence.

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1. Introduction

HfO_2 film is a promising high-*k* gate dielectric material to replace the traditional SiO_2 film in the advanced complementary metal–oxide–semiconductor field effect transistor (CMOSFET) devices [1,2]. In addition to a high dielectric constant, HfO_2 films have other advantages, such as a large band gap offset from that of Si and high thermal stability on silicon substrates [2–5]. Many methods for HfO_2 film deposition, such as sputtering, electron-beam evaporation, metal organic chemical vapor deposition, and atomic layer deposition (ALD), have recently been used [5–8]. Among these deposition methods, the ALD method is preferred for the scaled devices due to excellent thickness controllability and low thermal budget. A considerable amount of work has been investigated about ALD deposited HfO_2 dielectric films [8–10].

In order to improve HfO_2 dielectric film's properties, some studies reported that post-deposition plasma treatment, such as O_2, N_2 or NH_3 gas plasma, on the as-deposited HfO_2 film has positive improvement in electrical performance. However, these studies were usually performed with a fixed RF power [11–13]. Moreover, the HfO_2 gate dielectrics may encounter the plasma environments, such as the metal electrode deposition by sputtering during the device fabrication [14]. In the metal electrode sputtering process, the used RF power was usually higher than 50 W. To our best knowledge, little is known about how the RF power in the post-deposition plasma treatment affects physical and electrical properties of the deposited HfO_2 films, especially on the reliability impact. Therefore, the effects of the RF power in the post-deposition O_2 plasma treatment on the physical, electrical characteristics, and reliability of the HfO_2 films that were deposited using the ALD method were comprehensively investigated in this work.

2. Experiments

The HfO_2 films were deposited on a p-type Si (100) wafer with a resistivity of 1–10 Ω-cm. After performing the standard RCA cleaning process, the wafer was placed in a hot wall and traveling-wave type ALD chamber (Cambridge NanoTech. Inc. Savannah-100), which can process 4 in. wafer in one run. The HfO_2 film deposition uses TEMAHf ($Hf[NCH_3(C_2H_5)_2]_4$) and O_3 as the precursor and oxidant, respectively. N_2 was used as a carrier and purge gas. The O_3 delivery system generated 132 g/m^3 O_3 in N_2-diluted O_2. One ALD cycle consisted of a O_3 pulse, a N_2 purge, a TEMAHf pulse, and a N_2 purge. The oxidant and TEMAHf pulse lengths were 0.015 and 0.25 s, respectively. The N_2 purge time was 5 s. The deposition cycle was 80 cycles. HfO_2 films were deposited at a temperature of 200 °C. After the deposition, the HfO_2 films were post-treated with an O_2 plasma at 25 °C in a plasma-enhanced chemical vapor deposition chamber. The treatment time, pressure, and O_2 flow rate were 30 s, 4.0 Pa, and 100 sccm. The RF power was varied from 10 W to 200 W. After completing O_2 plasma treatment, a 500 nm-thick aluminum (Al) layer was deposited on top of the HfO_2 films using the evaporation method. This layer was then patterned by

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lithography and etching processes to form the gate electrode. Finally, all devices received backside aluminum deposition and thermal annealing at 425 °C to achieve a conductive contact for electrical measurements. The fabricated devices used for electrical and reliability measurements had an electrode area of 7854 μm^2.

The HfO_2 thin film thickness was measured using ellipsometry and these results were verified by high resolution transmission electron microscopy (HR-TEM) images. The thickness from ellipsometry measurement was calibrated using HRTEM measurement results and the error is within 5%. Surface morphologies and microstructures of the HfO_2 films were studied by atomic force microscopy (AFM) and X-ray diffractometry (XRD). The element profiles and chemical bonds of these samples were analyzed by Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS), respectively. Capacitance–voltage (*C–V*) and current–voltage (*I–V*) were measured at room temperature with an HP 4284A precision LCR meter at 1 MHz and with an HP 4156C semiconductor parameter analyzer, respectively. In reliability measurements, the conventional constant-voltage stress (CVS) test was used to determine the time to dielectric breakdown.

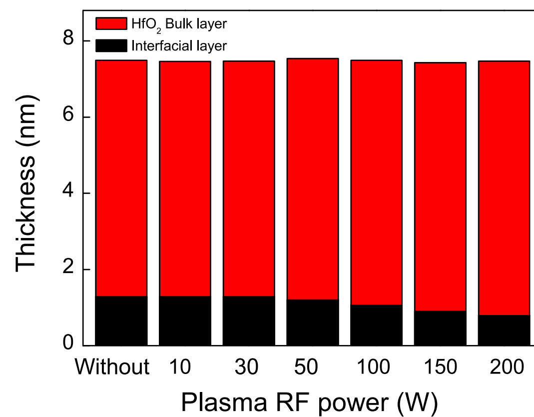

Fig. 2. Total thickness, the interfacial thickness, and bulk HfO_2 layer thickness as a function post-O_2 plasma RF power.

3. Results and discussion

Fig. 1 shows representative HR-TEM images of the as-deposited and post-O_2 plasma-treated HfO_2 films. All HfO_2 films comprise two layers — an interfacial layer (IL) and an upper HfO_2 layer. From TEM images, the interfacial layers and upper HfO_2 layers still remain amorphous and show no crystallization after O_2 plasma treatment, even when the treatment was performed at a 200 W RF power. XRD characterization (not shown) also showed no crystal peak regardless of the RF power in the O_2 plasma treatment, indicating that the HfO_2 film did not crystallize by post-deposition plasma treatment. Fig. 2 compares the averaged thicknesses of total HfO_2 film, the interfacial layer thickness, and the upper HfO_2 layer as a function of O_2 plasma power. The total thickness remains unchanged by the post-deposition O_2 plasma treatment and is not affected by the RF power. However, the thicknesses of the interfacial layer and the upper HfO_2 layer are dependent of the RF power. As the RF power is larger than 50 W, the thickness of the interfacial layer obviously decreases while that of the upper HfO_2 layer increases.

The surface roughness of the films was studied by AFM. The AFM images reveal that all HfO_2 films treated with various plasma powers are smooth without any indistinguishable features, as shown in Fig. 3(a). The root-mean-square (RMS) roughness of the HfO_2 films that were post-treated in an O_2 plasma was also calculated over a 2×2 cm^2 area from 5 sites, as presented in Fig. 3(b). The RMS roughness value of the as-deposited HfO_2 film is 0.22 nm and decreases with the RF power of the post-O_2 plasma treatment. Moreover, the variation of the RMS roughness values becomes smaller for the post-O_2 plasma-treated HfO_2 films with a higher RF power. The results indicate that the surface of the film becomes smoother and uniform after O_2 plasma treatment. A decreasing RMS roughness value by O_2 plasma treatment can result from the diffusion and mobility of the surface atoms by obtaining sufficient energy from O_2 plasma treatment [15] or the ion bombardment effect in the plasma environment.

XPS analysis was used to analyze the bonding structure and compositional changes at the bulk film surface and the interfacial layer of HfO_2/Si after post-O_2 plasma treatment with various RF powers. All measured spectra were calibrated by setting the C1s peak at 284.5 eV. Fig. 4(a) exhibits Hf 4f core level spectra at the surface of the post-O_2 plasma-treated HfO_2 films with various RF powers. All Hf 4f core level spectra have doublet peaks, corresponding to Hf $4f_{7/2}$ (peak at ~17.2 eV) and Hf $4f_{5/2}$ (peak at ~18.4 eV) which originate from Hf–O bonds [16]. The Hf 4f spectra also exhibit a peak shift to the lower binding energy with an increasing RF power of post-O_2 plasma and reach to a saturated binding energy as the RF power is raised to 50 W. The saturated binding energies in Hf 4f doublet peaks were 16.7 eV and 18.1 eV. The higher binding energy in Hf 4f peak for the HfO_2 film represents the O-deficient feature. A decreasing binding energy in the Hf 4f peak has been reported for the case that the resulting metal oxide film is fully oxidized which approaches to stoichiometric state [17]. Therefore, the oxygen vacancies can be repaired by post-O_2 plasma treatment. To investigate the chemical state at the HfO_2/Si interface, XPS spectra were detected after performing the Ar^+ etching process. Fig. 4(b) exhibits Hf 4f core level spectra at the interface of the post-O_2 plasma-treated HfO_2 films with various RF powers. The HfO_2 film without post-O_2 treatment also shows two double-peak components in the Hf 4f spectra, but both peaks shift to a higher binding energy, corresponding to the Hf 4f in $HfSi_xO_y$. Moreover, as the RF power of post-O_2 plasma treatment is increased to above 50 W, another peak at 14.8 eV

RF power	Without	10 W	30 W	50 W	100 W	150 W	200 W
TEM Image	6.21 nm	6.18 nm	6.19 nm	6.34 nm	6.43 nm	6.53 nm	6.68 nm
1nm	1.28 nm	1.28 nm	1.28 nm	1.2 nm	1.06 nm	0.9 nm	0.79 nm

Fig. 1. HR-TEM images of the as-deposited and post-O_2 plasma-treated HfO_2 films with various RF powers.

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