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Atomic layer etching of BeO using BCl_3/Ar for the interface passivation layer of III–V MOS devices

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ABSTRACT

BeO was investigated as the interface passivation layer (IPL) between a high-*k* dielectric and a III–V compound semiconductor substrate in metal-oxide-semiconductor (MOS) devices. One of the critical issues facing the fabrication of next generation MOS devices is the minimization of damage to the III–V semiconductor substrate during the etching of the thin IPL. In this study, atomic layer etching (ALET) was investigated for etching of BeO as the IPL on a GaAs substrate to control the etch depth precisely and to minimize the damage to the III–V semiconductor substrate. By using ALET to etch BeO, which uses BCl_3 as the adsorption gas for the formation of chloride compounds (Be–Cl and BCl–O) and Ar as the desorption gas for the removal of the chloride compounds, a self-limited, one-monolayer etch rate of about 0.75 Å/cycle was achieved with no increase of surface roughness and without change of surface composition. Moreover, under the BeO ALET conditions, which are able to precisely stop etching over the GaAs substrate after removing BeO, the exposed GaAs substrate showed surface composition and surface roughness similar to those of the as-received GaAs substrate.

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1. Introduction

High-*k* dielectrics on Si substrates must be extended to III–V compound semiconductor substrates because the latter with high electron mobility has received attention as channel materials for use in next generation metal-oxide-semiconductor (MOS) devices [1,2]. However, one of the main problems of III–V materials is that the compounds for which the materials are used are deficient of stable native oxides compared to Si, which are required to passivate the interface between the high-*k* dielectric and the substrate. To address this problem, various materials, such as Si, Ge, SiO_2, and Al_2O_3 etc., have been considered for the interface passivation layer (IPL) [3–5]. Recently, crystalline oxides, such as BeO, which has a smaller atomic radius (8.28 cm^3/mol) than that of Al_2O_3 (25.8 cm^3/mol), supporting its highly dense structure, have been studied largely because of their lack of crystallization induced grain boundary formation during post deposition annealing (PDA) [6,7].

As the critical dimension (CD) in IPL patterning is scaled down to tens of nanometers and below, plasma etching becomes more important because it can be used for anisotropic etching for accurate CD control. Unfortunately, due to the very low physical thickness of the IPL of about from 0.5 to 1 nm, it is difficult to remove with the minimal recess of the III–V compound semiconductor substrate. Therefore, a precise etch rate, instead of a high etch rate, and a very high etch selectivity between the IPL and the III–V compound semiconductor substrate are required. There have been many studies on the plasma etching of the IPL [8,9], however, these plasma IPL etchings tend to physically damage the etched surface of the III–V compound semiconductor substrate by creating the defects. Because of the energetic reactive ions used to make the anisotropic gate profiles, the desired electric characteristics of the III–V MOS device can be degraded [10–12].

Atomic layer etching (ALET) has been investigated as a technique for etching materials on the atomic scale without causing any physical and chemical damage to the substrate. ALET is a cyclic process similar to atomic layer deposition (ALD), but after

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one cycle, unlike ALD, which deposits one atomic layer, ALET etches one atomic layer. ALET consists of four steps: (1) the adsorption of reactive atoms/molecules on the surface, (2) the evacuation of the residual reactants, (3) the desorption of chemisorbed compounds on the surface by energetic Ar beam bombardment, and (4) the evacuation of the process chamber. The details of ALET can be found elsewhere [13,14].

In this article, ALET of BeO as the IPL on a III–V compound semiconductor substrate was studied because it may be the most suitable method for IPL patterning. The possibility of one monolayer etching per etch cycle and precise etch depth control of the very thin BeO layer with minimal GaAs substrate damage was also explored.

2. Experimental

BeO with the thickness of 25 Å was deposited on the GaAs substrate using a precursor ($Be(CH_3)_2$) and H_2O by atomic layer deposition (ALD) and patterned with a photoresist (PR) [15]. One cycle of the ALET studied was performed as follows: In the 1st step, BCl_3 gas was supplied to be adsorbed on the surface of BeO for 30 sec. In the 2nd step, the ALET system was evacuated to remove the unreactant BCl3 gas for 30 s. In the 3rd step, a low energy Ar beam from a beam source was applied for 125 s to desorb the chemisorbed compound materials on the BeO surface. In the final step, the ALET system was evacuated again for 30 s. (Currently, the process steps were not optimized, therefore, the process time takes a long time. Later, the process time will be decreased by optimizing the desorption time and evacuation time. In addition, the ion beam flux will be increased by optimizing ion beam source too.) For the adsorption of BCl_3 on the BeO surface, the BCl_3 flow rate was varied from 0 to 100 sccm. For the Ar beam source of the desorption step, a three-grid inductively coupled plasma (ICP) ion source operated with Ar gas was used to extract a parallel Ar energetic ion beam and, in front of the source, parallel reflecting plates were installed to obtain an Ar neutral beam (the details on the neutralization of an Ar ion beam can be found elsewhere [16,17]. For the desorption of chlorides on the BeO surface by the Ar beam, the following Ar beam condition was applied: Ar gas flow rate: 50 sccm; 13.56 MHz ICP power: 300 W; 1st grid voltage, varied, 50–150 V; 2nd grid voltage: −250 V; and 3rd grid voltage: grounded.

After the ALET of BeO, the etch depth was measured using a step profile meter (Tencor Instrument, Alpha Step 500). The measured etch depth (Å) was divided by the total number of cycles to estimate the etch rate (Å/cycle). Atomic force microscopy (AFM, Thermomicroscope, CP Research) was used to measure the surface roughness. X-ray photoelectron spectroscopy (XPS, Thermo VG, MultiLab 2000, Mg Kα source) was used to measure the surface composition of the etched BeO and GaAs substrate after the etching of BeO. To measure the AFM root mean square (RMS) roughness and atomic ratio, BeO was etched for 25 cycles.

3. Results and discussion

For the ALET of BeO, BCl_3 needs to fully covered on the BeO surface during the adsorption step for the formation of one monolayer of the chloride compounds (Be–Cl and BCl–O), and then the monolayer needs to be removed completely by energetic Ar beam irradiation without sputter etching of the exposed BeO beneath the monolayer. To remove only the chloride compounds without sputter etching of BeO, an adequate energy of the Ar beam needs to be supplied. When BCl_3 is not adsorbed on the BeO surface, the bonding strength between Be and O in the 1st layer (E_a) and that in the 2nd layer (E_b) are the same. However, after the adsorption of BCl_3, the E_a would be lower than E_b because the electron moves from Be–O to the Be–Cl and BCl–O bonds [18]. This transfer enables the 1st layer to be broken at a lower energy than the energy needed to break the 2nd layer, so one monolayer etching is carried out during the ALET. Therefore, even though the enough Ar beam dose during the desorption step is sufficient, if the transferred energy of the Ar beam to the chemisorbed 1st layer (E_n) is equal to or lower than E_a, that is, $E_n \leqslant E_a$. Under these condition, a partial layer etching of compounds is obtained, and so, an etch rate of less than one monolayer per cycle is obtained. If the transferred energy of the Ar beam is $E_a < E_n < E_b$, the ALET condition results in one monolayer etching per cycle. However, if $E_b < E_n$ due to the high energy of the Ar beam, after the removal of chemisorbed 1st layer, the 2nd layer under the compound layer, that is BeO, is sputter etched; therefore, an etch rate of greater than one monolayer per cycle is obtained. Therefore, during the desorption step, it is important that the appropriate Ar beam energy is used to desorb only the chemisorbed compounds on the BeO surface.

To determine the appropriate energy for the ALET condition of BeO, the etch rate (Å/cycle) of BeO was measured as a function of Ar beam energy (1st grid voltage) during cyclic etching with and without the supply of BCl_3 gas during the adsorption step. To form one monolayer of chemisorbed chlorides (Be–Cl, and BCl–O) during the adsorption step, 100 sccm of BCl_3 gas flow was supplied. When BCl_3 was not supplied during the cyclic etching, BeO was etched only by sputtering; however, when BCl_3 was supplied during the adsorption step, BeO was etched after the chemisorbed species (Be–Cl and BCl–O) were removed by Ar beam bombardment during the desorption step.

Fig. 1 shows the etch rate (Å/cycle) of BeO measured as a function of 1st grid voltage of the desorption step from 50 to 150 V with and without the supply of BCl_3 gas during the adsorption step. As shown in the figure, in the case when BCl_3 is not supplied during the adsorption step, the etch rate curve is divided into two regions: for the voltage range from 0 to about 130 V (regions I + II), no or negligible etching of BeO is observed and, for voltages higher than about 130 V (region III), the etch rate starts increasing with increasing voltage. These results indicate that about 130 eV is the threshold energy (E_b) for BeO sputtering. On the other hand, in the case when BCl_3 is supplied during the adsorption step, the etch rate is divided into three regions: for voltages up to about 120 V (region I), the etch rate is increased with increasing voltage and, for the voltage range from about 120 to about 130 V (region II), the etch rate is almost saturated, and, for voltages higher than 130 V (region III), the etch rate again increases with increasing voltage. Region I represents partial BeO etching per cycle due to the insufficient energy transfer by the Ar beam supplying energy

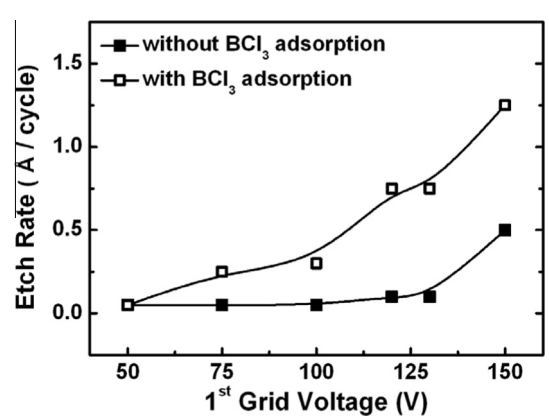

Fig. 1. Etch rate (Å/cycle) of BeO measured as a function of 1st grid voltage of the desorption step from 50 to 150 V with/without the supply of BCl_3 gas during the adsorption step.

E_n equal to or lower than E_a ($E_n \leqslant E_a$), as mentioned earlier. However, in region II, by supplying energy E_n between E_a and E_b to the substrate, the BeO etch rate remains constant, one monolayer per cycle, because the chemisorbed 1st layer is removed only during the desorption step without sputter etching of the BeO layer exposed after the removal of the chemisorbed 1st layer. The differences between two curves are the largest in this region, indicating that the ALET condition is obtained. In region III, after the removal of the chemisorbed chloride 1st layer, the exposed 2nd BeO layer is further sputtered due to E_n being higher than both E_a and E_b.

In fact, to obtain one monolayer etching per cycle, in addition to supply Ar energy E_n between E_a and E_b with a sufficient Ar dose to remove all chemisorbed materials during the desorption step, one complete monolayer chemisorbed layer should be formed on the BeO surface during the adsorption step by BCl_3. In the Langmuir isotherm, the adsorption of reactive BCl_3 gas by a single chemisorbed layer on the BeO surface can be expressed as

$$BCl_{3(g)} + BeO \xrightarrow{k_1} BeCl_{x(ad)} + BCl_yO_{(ad)}$$

where k_1 is the adsorption rate constant. Because its surface coverage (Θ) is dependent on the gas flow rate (F), which is related to the gas pressure (P) at the same pumping speed, the surface coverage of $BeCl_x$–BCl_yO bonds θ_{BeCl_x/BCl_yO} is expressed as

$$\theta_{BeCl_x/BCl_yO} = \frac{\sqrt{k_1 P_{BCl_s}}}{1 + \sqrt{k_1 P_{BCl_s}}}$$

where P_{BCl_s} is the BCl_3 pressure. Therefore, θ_{BeCl_x}/BeC_yO is dependent on the P_{BCl_s}. If the supplied P_{BCl_s} is lower than the critical pressure for one monolayer coverage, $\sqrt{k_1 P_{BCl_s}}$ is smaller than 1 and θ_{BeCl_x}/BeC_yO could be approximately represented as $\theta_{BeCl_x}/BeC_yO \approx \sqrt{k_1 P_{BCl_s}}$. On the other hand, if the supplied P_{BCl_s} is higher than the critical pressure, $\sqrt{k_1 P_{BCl_s}}$ is higher than 1 and θ_{BeCl_x}/BeC_yO could be approximately represented as $\theta_{BeCl_x}/BeC_yO \approx 1$. Therefore, it is an important factor for the self-limited one monolayer etching, and enough BCl_3 flow rate needs to be supplied during the adsorption step to obtain one monolayer adsorption on the BeO surface. The adsorbed species are desorbed on the BeO surface during the desorption step through the following equation.

$$BeCl_{x(ad)} + BCl_yO_{(ad)} \xrightarrow{k_1} BeCl_{x(g)} + BCl_yO_{(g)}$$

where k_2 is the adsorption rate constant [19].

To investigate the required BCl_3 flow rate, that is, the BCl_3 pressure on the BeO etch rate for the ALET condition, the BeO etch rate (Å/cycle) was studied as a function of BCl_3 flow rate during cyclic etching. Fig. 2 shows the etch rate (Å/cycle) of BeO and the root mean square (RMS) roughness of the etched BeO surface measured using AFM as a function of BCl_3 gas flow rate from 0 to 100 sccm during adsorption. For the desorption, +125 V of 1st grid voltage was applied to the Ar beam source and the Ar beam was applied for 125 s for the desorption of all chemisorbed materials on the BeO surface. As shown in the figure, the etch rate of BeO increased with BCl_3 gas flow rate up to 50 sccm and saturated with further increase of BCl_3 gas flow rate. The saturated etch rate of BeO was about 0.75 Å/cycle, which was consistent with the deposition rate of BeO by ALD [20]. When the surface roughness was measured after etching for 25 cycles, the surface rough initially increased with the increase of BCl_3 flow rate from 3.7 Å for 0 sccm of BCl_3 flow rate to 5.57 Å for 25 sccm; however, as the ALET condition was reached by supplying higher BCl_3 flow rate, the surface roughness decreased and finally remained similar to that of the reference (as-received BeO). The initial increase of surface roughness with the increase of BCl_3 flow rate was related to the repeated partial layer removal of the BeO surface per etch cycle due to the partial coverage of adsorbed BCl_3 on the BeO surface. However, as soon as the BeO surface was fully covered by BCl_3 and one monolayer of the chemisorbed compounds was formed on the BeO surface, the surface roughness decreased and remained similar to that of as-received BeO because of the complete removal of the surface chemisorbed monolayer per etch cycle.

When the ALET condition is used, the etch depth per cycle should be maintained the same throughout the etching period to obtain an accurate etch depth within the atomic scale error range. Fig. 3 shows the etch depth (Å) and etch rate (Å/cycle) of BeO and RMS roughness (Å) of the etched BeO surface measured as a function of the number of etch cycles for the ALET condition. As the ALET condition, 100 sccm of BCl_3 gas flow rate for 30 s (for the adsorption), Ar beam with +125 V of 1st grid voltage for 125 s (for the desorption), and the evacuation time for 30 s between adsorption and desorption were used. As shown in the figure, the etch depth (Å) increased exactly linearly with increasing number of etch cycles when the same BeO etch rate was maintained throughout the etch period. In addition, the RMS roughness of the etched BeO surface also remained constant, similar to that of as-received BeO, regardless of the number of etch cycles.

During conventional etching of compound materials, the composition of the surface layer is generally changed due to the differences in the vapor pressures of the chemisorbed compounds. Fig. 4 shows the atomic percentages of Be and O and the Be/O ratio of the etched BeO surface measured by XPS for the as-received BeO, the BeO

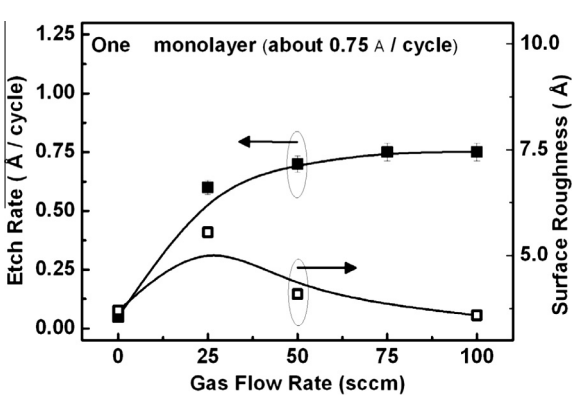

Fig. 2. Etch rate (Å/cycle) of BeO and the RMS roughness of the etched BeO surface measured as a function of BCl_3 gas flow rate from 0 to 100 sccm during the adsorption by AFM. For the desorption, 125 V of 1st grid voltage was applied to the Ar beam source and the Ar beam was applied for 125 s for the desorption of all chemisorbed materials on the BeO surface.

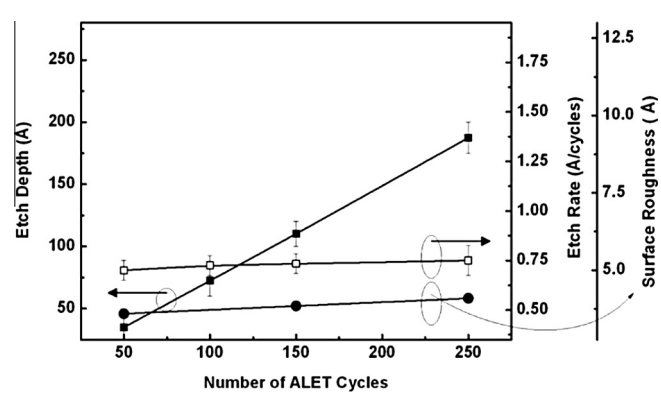

Fig. 3. Etch depth (Å) and etch rate (Å/cycle) of BeO and RMS roughness (Å) of the etched BeO surface measured as a function of the number of etch cycles for the ALET one monolayer etching condition, which consisted of 100 sccm of BCl_3 gas flow rate for 30 s (for the adsorption), Ar beam with 125 V of 1st grid voltage for 125 s (for the desorption), and evacuation time for 30 s between the adsorption and the desorption.

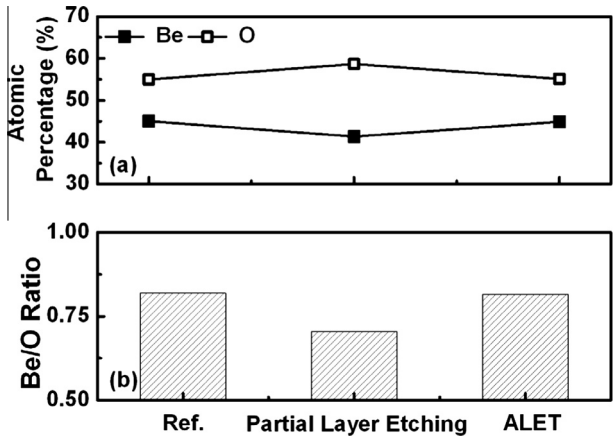

Fig. 4. Atomic percentages of O and Be? (a) and the Be/O ratio (b) of the etched BeO surface measured by XPS for the as-received BeO, the BeO etched in the partial layer etching condition ($\Theta_{Be-Cl/BCl-O} < 1$, BCl_3 25 sccm), and the BeO in the ALET condition ($\Theta_{Be-Cl/BCl-O} = 1$, BCl_3 100 sccm). Other process conditions were the same as those in Fig. 2.

etched in the partial layer etching condition ($\Theta_{Be-Cl}/_{BCl-O} < 1$, BCl_3 25 sccm), and the BeO etched in the ALET condition ($\Theta_{Be-Cl}/_{BCl-O} = 1$, BCl_3 100 sccm). Other process conditions were the same as those in Fig. 2. They were measured at the take-off angle of 25°, which can be estimated to be within about 10 Å of the surface layer. As shown in the figure, the percentage of O was a little higher than that of Be for the as-received BeO; therefore, the ratio of Be/O was lower than 1.0. After etching in the partial layer etching condition, similar to the conventional etching condition, the surface composition of the BeO surface changed to more O-rich and the Be/O ratio was further decreased possibly due to the preferential removal of Be on the surface during partial layer etching. However, in the case of etching in the ALET condition, the composition of the BeO surface was maintained similar to that of the as-received BeO because one monolayer was fully etched on the BeO surface during each etch cycle. It is believed that, for the fabrication of a III–V MOS device with BeO as the IPL, if the IPL layer is etched in the ALET condition, the composition of the IPL edge will be maintained the same as that of the as-received BeO after the IPL etching;, therefore, the possible leakage current of the MOS device at the edge of the IPL due to the change of surface composition can be minimized.

During III–V MOS device fabrication, the unmasked IPL area should be etched in full and the GaAs substrate should be exposed to form source and drain contacts by over-etching. Using the above BeO etching conditions, the GaAs substrate was exposed, and the roughness and the As/Ga ratio of the exposed GaAs surface were investigated. Fig. 5 shows the RMS roughness (Å) of the etched GaAs substrate surface measured by AFM for the partially layer etching condition ($\Theta_{Be-O}/_{BCl-O} < 1$, BCl_3 = 25 sccm) and the ALET condition ($\Theta_{Be-O}/_{BCl-O} = 1$, BCl_3 = 100 sccm) in Fig. 2. As the reference, the roughness and As/Ga ratio of the as-received GaAs were also measured. As shown in the figure, in the case of the partial layer etching condition, the surface roughness of the GaAs substrate surface increased compared to that of the as-received GaAs surface and the composition also became more Ga-rich due to the repeated partial layer removal per etch cycle; however, for the BeO ALET condition, the roughness and As/Ga ratio were similar to those of the as-received GaAs. Therefore, it is believed that the BeO ALET can minimize the damage to the GaAs substrate, which might be caused by the increased roughness and the changed composition of the exposed GaAs substrate surface.

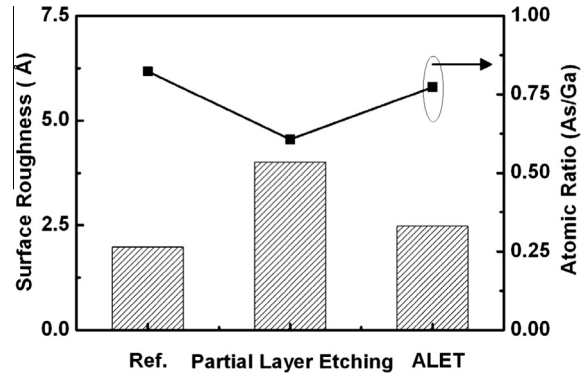

Fig. 5. RMS roughness (Å) of the etched GaAs substrate surface measured by AFM for the partially layer etching condition ($\Theta_{Be-O/BCl-O} < 1$, BCl_3 25 sccm) and for the ALET condition ($\Theta_{Be-O/BCl-O} = 1$, BCl_3 100 sccm) in Fig. 2. As the reference, the roughness and As/Ga ratio of the as-received GaAs were also measured.

4. Conclusions

In this article, the ALET of BeO as the IPL on a III–V compound semiconductor substrate was studied using BCl_3 as the adsorption gas for the formation of chloride compounds (Be–Cl and BCl–O) and Ar as the desorption gas for the removal of the chloride compounds. The ALET condition, that is, the etching of one monolayer per etch cycle, was observed by providing BCl_3 reactive gas at a pressure higher than the critical gas pressure for the formation of one chemisorbed monolayer on the surface and by providing Ar gas for the removal of all the chemisorbed species during the desorption step. Using the ALET technique, a self-limited one monolayer BeO etch rate of about 0.75 Å/cycle was achieved with no increase of surface roughness and no change in surface composition during the etching process. Surface roughness and compositional change of the exposed GaAs substrate surface were also minimized.

Acknowledgments

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