

Etching Characteristics of ZnO and Al-Doped ZnO in Inductively Coupled Cl₂/CH₄/H₂/Ar and BCl₃/CH₄/H₂/Ar Plasmas

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(Received November 11, 2007; revised June 2, 2008; accepted June 25, 2008; published online August 22, 2008)

ZnO and Al-doped ZnO (AZO) were etched in Cl₂/CH₄/H₂/Ar (Cl₂-based) and BCl₃/CH₄/H₂/Ar (BCl₃-based), inductively coupled plasmas (ICPs) and their etching characteristics were compared by varying the Cl₂/(Cl₂ + CH₄) and BCl₃/(BCl₃ + CH₄) flow ratios, top electrode power and dc self-bias voltage (V_{dc}). The etch rates of both ZnO and AZO layers were higher in the Cl₂-based chemistry than in the BCl₃-based chemistry. The AZO and ZnO etch rates were increased and decreased, respectively, with increasing Cl₂ or BCl₃ flow ratio. Optical emission measurements of the radical species in the plasma and surface binding states by optical emission spectroscopy (OES) and X-ray photoelectron spectroscopy (XPS), respectively, indicated that, with increasing Cl₂ or BCl₃ flow ratio; the effective removal of Al in the AZO enhanced the AZO etch rate, whereas the reduced removal of Zn by the Zn(CH_x)_y products reduced the ZnO etch rate.

[DOI: 10.1143/JJAP.47.6960]

KEYWORDS: TCO, ZnO, Al-doped ZnO (AZO), plasma etching, inductively coupled plasma (ICP)

1. Introduction

The transparent conductive oxide (TCO) layer is used as an electrode that is transparent to visible light. The increasing use of TCO films has focused research and development on finding alternative and cheaper TCO materials such as Al-doped ZnO (AZO) because of the high cost of indium tin oxide and the tendency for its properties to degrade upon exposure to plasma.^{1,2} AZO has good transmittance in the visible light and infrared range, electrical conductivity, processibility at reduced temperature and low cost. Recently, extensive research has been conducted on ZnO and AZO for large-area display devices.³

Research on the dry etching of ZnO in inductively coupled plasmas (ICPs)^{4–13} and electron cyclotron resonance plasmas¹⁴ has been reported by several research groups. The ICP etching of ZnO has been investigated with various plasma parameters, such as different gas combinations (CH₄/H₂/Ar,^{4,6,7,11,13} BCl₃/Cl₂/Ar,^{5,12} BCl₃/Ar,^{10,12} BCl₃/CH₄/H₂,^{8,10} and Cl₂/Ar^{6,9,12}), and different electrode power levels.^{4,5,9,10} The typical etch by-products formed during dry etching processes utilizing Cl₂ or BCl₃ gas are presumably ZnCl₂⁹ and B-containing compounds such as boron oxychloride (BOCl), BO₂, and (BOCl)₃.^{5,8} Due to the low vapor pressure of ZnCl₂ (1 Torr at 428 °C),^{4,15} the ZnO etching in the chlorine-containing plasma requires a significantly energetic ion bombardment for the effective removal of the etch by-products. The formation of the etch by-product, Zn(CH_x)_y, during the doped-ZnO etching using CH₄-containing plasma led to an effective etch process for ZnO and doped-ZnO layers.^{4,11,13} Our group has also shown that use of CH₄ gases was effective in obtaining infinitely high etch selectivities of the ZnO and AZO layers to the photoresist (PR).^{11,13} In this case, the high degree of polymerization leads to the formation of CH_x polymer on the chamber walls, which necessitates alleviating the

polymerization during CH₄/H₂/Ar ICP etching. The effect of Cl₂ and BCl₃ addition to the CH₄/H₂/Ar chemistry is therefore of interest in reducing the polymer formation during etching.

In this study, the etch characteristics of AZO and ZnO thin films in Cl₂/CH₄/H₂/Ar (Cl₂-based) and BCl₃/CH₄/H₂/Ar (BCl₃-based) ICPs and the effects of Cl₂ and BCl₃ gases in the CH₄-based etch plasmas were investigated. The Cl₂/(Cl₂ + CH₄) and BCl₃/(BCl₃ + CH₄) flow ratios affected the etch characteristics of the ZnO and AZO layers differently due to densities in the plasma and the presence of aluminum in the AZO layer.

2. Experiment

A modified, commercial 8-in. ICP conductor etcher with a 3.50-turn spiral copper coil on the top of the 8.5 L chamber separated by a 1 cm-thick quartz window was used in this experiment.¹² A schematic of the ICP etch system used in the present experiment is depicted elsewhere.¹⁶ The radio frequency power of 13.56 MHz was applied to the top electrode coil to induce the ICP. A bottom bias electrode power of 13.56 MHz was applied to the substrate holder to induce the dc self-bias voltage (V_{dc}) to the wafer.

AZO(0002) (Al = 2 wt %) and ZnO(0002) thin films with a thickness of 200–300 nm were prepared on 10 × 10 mm² glass wafer substrates and then underwent photolithographic patterning of a positive PR mask (AZ 7220) on AZO/glass and ZnO/glass. The profile angle of the patterned PR was $\cong 75^\circ$ and the PR thickness was about 1.8 μm . The samples were fixed on an 8-in. silicon wafer placed on the substrate holder using a heat-conductive paste, DC 340 (Dow Corning). The substrate holder temperature during etching was kept at 18 °C by circulating cooling water using a chiller, which prevented burning of PR on the Si wafer. During the experiments, the working pressure was kept at 15 mTorr.

The AZO and ZnO etch rates were measured by partially etching the AZO and ZnO layers with the PR mask using

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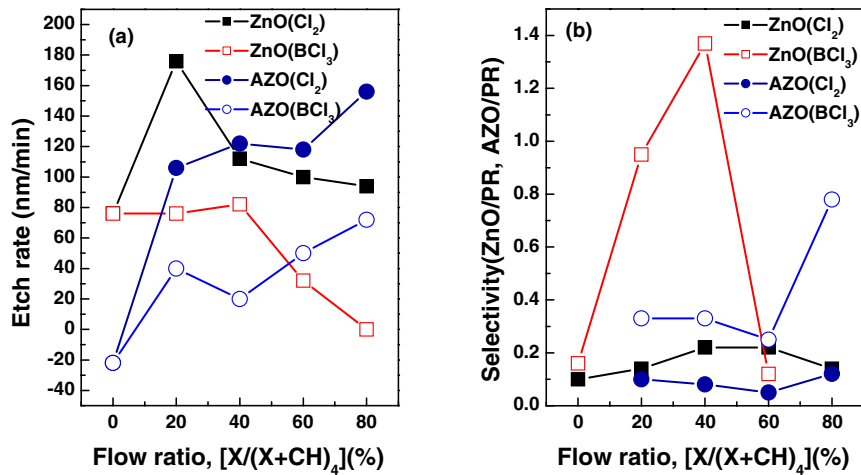


Fig. 1. (Color online) Etch (a) rates and (b) selectivity of the ZnO and AZO layers to PR as a function of the $X/(X + \text{CH}_4)$ gas mixture ratio ($X = \text{Cl}_2$ and BCl_3) at a fixed total gas of 100 sccm flow, top power of 500 W, V_{dc} of -250 V, operating pressure of 15 mTorr, etch time of 60 s, Ar flow rates of 20, and H_2 flow rate of 55 sccm.

field emission scanning electron microscopy (FE-SEM). A multi-channel optical emission spectrometer (Ocean Optics) with the spectral range of 270–900 nm was used to analyze the optical emission from the radicals in the plasma. The surface binding states and composition of the etched layers were investigated using an Auger electron spectroscopy and X-ray photoelectron spectroscopy system (AES-XPS; ESCA2000). The Mg $K\alpha$ source provided non-monochromatic X-rays at 1253.6 eV.

3. Results and Discussion

Figure 1(a) shows the etch rates of the ZnO and AZO thin films etched by varying the $\text{Cl}_2/(\text{Cl}_2 + \text{CH}_4)$ and $\text{BCl}_3/(\text{BCl}_3 + \text{CH}_4)$ flow ratios from 0 to 80% at a fixed H_2/Ar flow rate of 55 sccm/20 sccm and a total flow rate of 100 sccm for 60 s. The top electrode power and V_{dc} were fixed at 800 W and -250 V, respectively. The ZnO and AZO etch rates were faster in the Cl_2 -based plasmas than in the BCl_3 -based plasmas. The ZnO etch rate was increased and then decreased with increasing the Cl_2 flow rate in the Cl_2 -based plasmas. The ZnO etch rate remained almost unchanged as the BCl_3 flow ratio was increased up to 40% but then decreased as the BCl_3 flow ratio exceeded 40% in the BCl_3 -based plasmas. However, the AZO etch rates were increased with increasing Cl_2 and BCl_3 flow ratios. Due to contrasting tendencies in the etch rates of the ZnO and AZO layers according to the changing gas flow ratios, the ZnO etch rate was higher than the AZO etch rate at low flow ratios but lower at high flow ratios exceeding 60%. Figure 1(b) shows the etch selectivity values of the ZnO and AZO layers to the PR. Selectivity values could not be obtained for ZnO etched in 80% BCl_3 chemistry or for AZO etched in 0% Cl_2 and BCl_3 chemistries. The etch selectivities of ZnO and AZO in BCl_3 -based plasmas were generally higher than those in Cl_2 -based plasmas.

Figures 2(a) and 2(b) show the cross-sectional FE-SEM images of the ZnO and AZO etched at the $\text{Cl}_2/(\text{Cl}_2 + \text{CH}_4)$ gas mixing ratios of 20 and 80%, respectively. The top electrode power, V_{dc} , and etch time were 800 W, -250 V, and 2 min, respectively. The profile angle of both samples was $\cong 70^\circ$. The profiles of the ZnO and AZO layers were

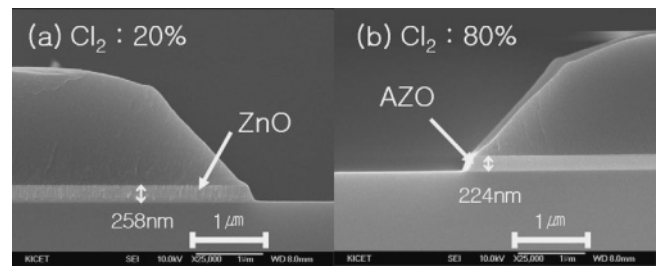


Fig. 2. FE-SEM images of the etched (a) ZnO at 20% Cl_2 , (b) AZO at 80% Cl_2 (etch conditions; ICP power: 800 W, dc self-bias voltage: -250 V, total gas flow rate: 100 sccm, H_2/Ar flow rates: 55/20 sccm, etching time: 2 min).

sloped due to the sloped nature of the patterned PR in our experiment. Optical emission spectroscopy (OES) of the species in the plasma was measured as a function of the gas flow ratios during etching in order to investigate any correlation between the relative change in the emission intensities of the species in the plasma and the etch rate variations. The optical emission intensity ratios relative to the Ar atom (750.92 nm) of the CH radical (431.35 nm), Cl radical (486.41 nm), Al atom (288.43 nm), and Zn atom (772.45 nm) ($[\text{CH}]/[\text{Ar}]$, $[\text{Cl}]/[\text{Ar}]$, $[\text{Al}]/[\text{Ar}]$, and $[\text{Zn}]/[\text{Ar}]$) were obtained and are shown in Fig. 3. As shown in Figs. 3(a) and 3(b), the $[\text{Zn}]/[\text{Ar}]$ and $[\text{CH}]/[\text{Ar}]$ emission intensity ratios during ZnO etching were decreased with increasing Cl_2 and BCl_3 gas flow ratios while the $[\text{Cl}]/[\text{Ar}]$ emission intensity ratio was increased. As shown in Figs. 3(c) and 3(d), however, the $[\text{Al}]/[\text{Ar}]$, $[\text{Zn}]/[\text{Ar}]$ and $[\text{Cl}]/[\text{Ar}]$ emission intensity ratios during AZO etching were increased with increasing Cl_2 and BCl_3 gas flow ratios while the $[\text{CH}]/[\text{Ar}]$ emission intensity ratio was decreased.

The decrease in the $[\text{Zn}]/[\text{Ar}]$ emission intensity ratio with increasing Cl_2 and BCl_3 flow ratios using ZnO etching and the increase in the $[\text{Zn}]/[\text{Ar}]$ and $[\text{Al}]/[\text{Ar}]$ emission intensity ratios during AZO etching are consistent with the etch rate variation of the ZnO and AZO layers with increasing Cl_2 and BCl_3 flow ratios (Fig. 1). In the case of AZO etching, it was previously shown that Al doping in

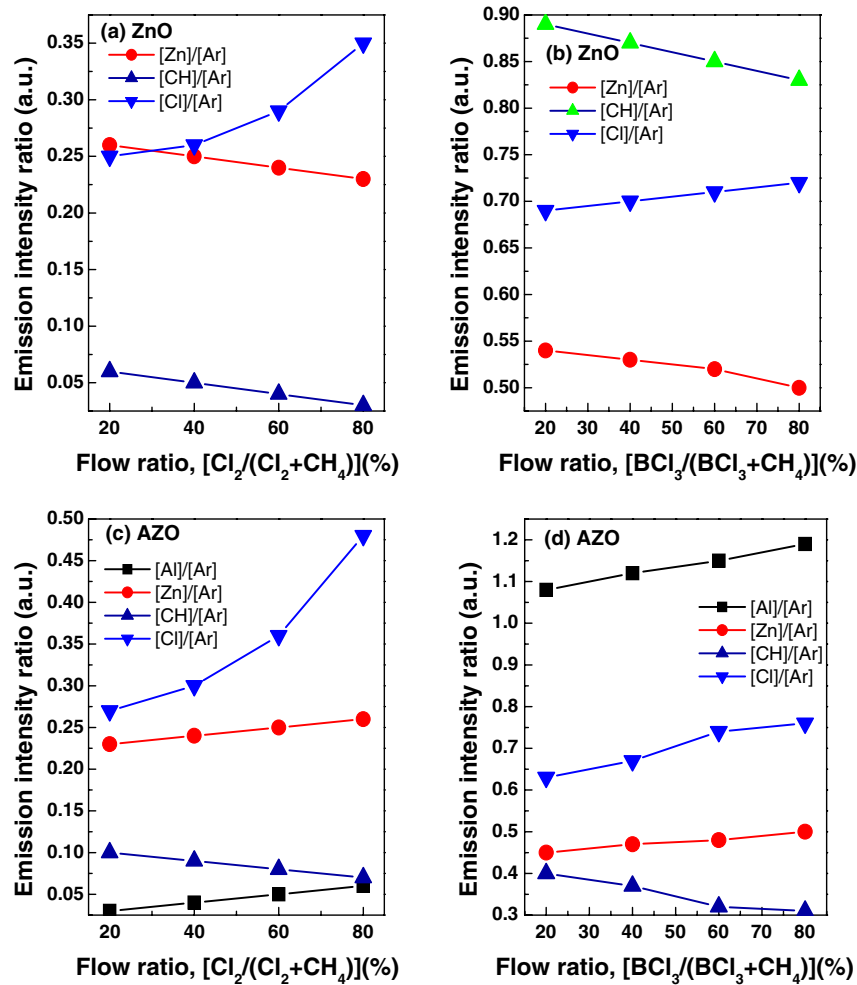


Fig. 3. (Color online) Optical emission intensities of the species during etching of (a) Cl_2 -etched ZnO, (b) Cl_2 -etched AZO, (c) BCl_3 -etched ZnO and (d) BCl_3 -etched AZO measured by OES as a function of $\text{Cl}_2/(\text{Cl}_2 + \text{CH}_4)$ and $\text{BCl}_3/(\text{BCl}_3 + \text{CH}_4)$ gas flow ratios (etch conditions; ICP power: 500 W, dc self-bias voltage: -250 V, total gas flow rate: 100 sccm, H_2/Ar flow rates: 55/20 sccm).

ZnO decreased the ZnO etch rate in the CH_4 -based plasmas due to the lower volatility of Al organic compound by-products compared to that of Zn organic by-products.¹³⁾ For example, trimethylaluminum $[\text{Al}(\text{CH}_3)_3]$ has a higher boiling point of 130°C (at 760 Torr) than that of 46°C for dimethylzinc $[\text{Zn}(\text{CH}_3)_2]$.¹⁷⁾ The OES data (Fig. 3) indicated that adding Cl_2 or BCl_3 in the $\text{CH}_4/\text{H}_2/\text{Ar}$ plasma enhanced the removal rate of Al atoms on the AZO surface by forming AlCl_x by-products and thereby enhanced the AZO etch rate. In the case of pure ZnO etching, the ZnO etch rate seems to be determined by a balance between Cl and CH_x radicals in the plasmas. At higher Cl_2 or BCl_3 flow ratio, the reduced formation of $\text{Zn}(\text{CH}_x)_y$ by-products due to lessened CH_x radicals in the plasma compared to the increased formation of ZnCl_2 and B-containing etch by-products probably exerted a bigger impact on decreasing the ZnO etch rate.

The surface composition and chemical binding states of the etched sample surfaces were examined by XPS for detailed examination of the effect of the gas flow ratios. To prepare the samples for XPS measurement, the etch time, top electrode power and V_{dc} were kept constant at 60 s, 800 W, and -150 V, respectively, while Cl_2 and BCl_3 flow ratios of 20% were used for ZnO etching and of 80% for AZO etching. The obtained Zn 2p and O 1s XPS spectra are

shown in Fig. 4. The peak intensities of the Al 2p, B 1s, and Cl 2p spectra were negligibly small for etched AZO samples (not shown).

After etching, the peak positions of Zn 2p_{3/2} (1021.90 eV) and 2p_{1/2} (1044.79 eV) of the unetched ZnO surface and of Zn 2p_{3/2} (1021.52 eV) and 2p_{1/2} (1044.69 eV) of the unetched AZO surface were shifted toward higher binding energies by $\cong 0.7$ eV, as compared to those from the unetched ZnO and AZO surfaces, as shown in Fig. 4(a). This shift in the binding energy indicates a chemical reaction of Zn with Cl and CH_x radicals, resulting in the formation of by-products such as ZnCl_2 and $\text{Zn}(\text{CH}_x)_y$ on the surface.^{11,13,15)}

The O 1s spectra in Fig. 4(b) showed that the intensities of the first (530.3 eV, attributed to Zn–O bonds) and second (532.1 eV, attributed to O–H bonds) peaks were reversed for the data obtained from the bare samples and etched samples. Here, the shoulder peak indicated as O–H from the as-deposited sample was related to the formation of Zn–OH bonds due to surface contamination of the ZnO by the hydroxyl groups due to air exposure.¹⁸⁾ With the addition of Cl_2 and BCl_3 gases, the Zn–O peak intensity became negligible, which indicated a dramatic increase in the Zn–Cl and Zn– CH_x bondings as a result of the chemical reaction of

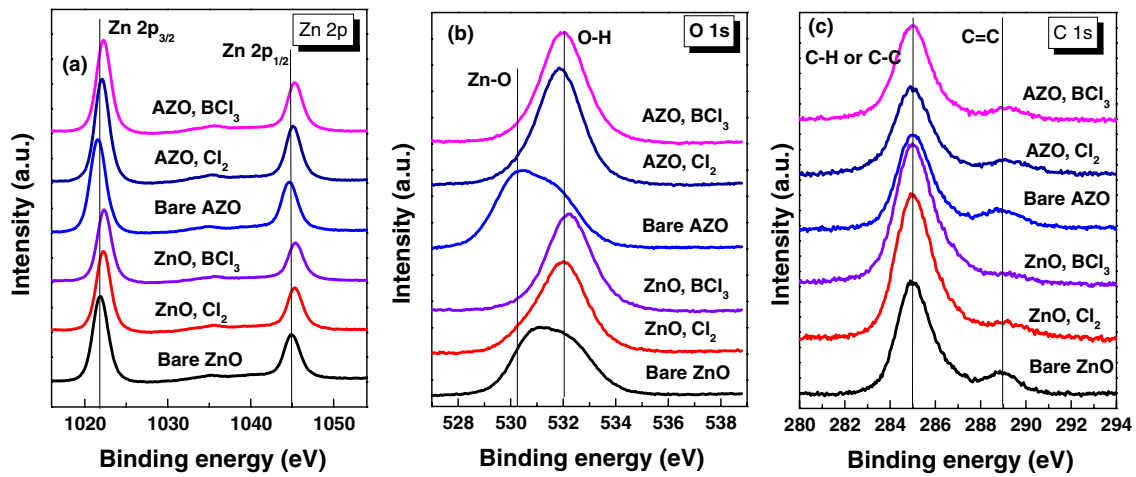


Fig. 4. (Color online) XPS spectra of (a) Zn 2p, (b) O 1s, and (c) C 1s (etch conditions; etch time: 60 s, top electrode power: 800 W, dc self-bias voltage: -250 V, Cl_2 and BCl_3 flow ratios for ZnO etching: 20%, Cl_2 , BCl_3 flow ratios for AZO etching: 80%).

Cl and CH_x radicals with the ZnO and AZO surfaces. For the etched samples, the intensities of the O–H peaks increased, presumably due to the increased formation of $\text{Zn}(\text{OH})_2$ in the air and possibly due to O–Cl bond formation, although that was not demonstrated here.

The C 1s spectra in Fig. 4(c) show that the intensities of the first (285 eV, attributed to C–H or C–C bonds) and second (288.9 eV, attributed to C=C bonds) peaks were not increased for the etched samples compared to the bare samples. This observation implied the effective removal of carbon species from the surface by the formation of etch by-products such as the Zn-organic compound, $\text{Zn}(\text{CH}_x)_y$. It is probable that the Zn–O bonds in the ZnO and AZO thin films were broken and effectively removed by the CH radicals.⁴⁾

The atomic concentration data obtained from the XPS spectra (Table I) indicated decreased concentrations of Zn, O and Al ([Zn], [O], and [Al]) after etching but an increased concentration of Cl ([Cl]) compared with the bare ZnO and AZO. The concentration ratios of oxygen to zinc, [O]/[Zn], for the etched samples were increased compared to those of the bare samples and were higher for the samples in the etched BCl_3 -based plasmas than in the Cl_2 -based plasmas. The lower [O]/[Zn] values in the Cl_2 -based plasmas might be correlated with the higher ZnO and AZO etch rates in the Cl_2 -based plasmas than in the BCl_3 -based plasmas. The effective removal of oxygen atoms in the ZnO and AZO layers by chemical reaction of the oxygen atoms with the carbon atoms in the definition (a-C:H) layer increased etch rates, probably because the effective removal of oxygen atoms can be accompanied by the enhanced chemical reaction of Zn with Cl and CH_x to form ZnCl_x and $\text{Zn}(\text{CH}_x)_y$ etch by-products, respectively. The lower [C]/[Zn] concentration ratios in the Cl_2 -based plasmas also explained the faster carbon consumption by the oxygen atoms in the Cl_2 -based plasmas than in the BCl_3 -based plasmas.

Figure 5 shows the AZO and ZnO etch rates as a function of V_{dc} at the $\text{Cl}_2/(\text{Cl}_2 + \text{CH}_4)$ gas mixing ratio of 20 and 80%, respectively, and the top electrode power of 800 W for 60 s. Etching was performed by varying the dc self-bias voltage from -100 to -250 V. The AZO and ZnO etch rate

Table I. Surface atomic concentration ratios of the bare and etched, ZnO and AZO layers determined from XPS spectra (etch conditions; etch time: 60 s, top electrode power: 800 W, dc self-bias voltage: -250 V, Cl_2 and BCl_3 flow ratios for ZnO etching: 20%, Cl_2 and BCl_3 flow ratios for AZO etching: 80%).

Concentration	ZnO			AZO		
	Bare	Cl_2 (20%)	BCl_3 (20%)	Bare	Cl_2 (80%)	BCl_3 (80%)
[O]/[Zn]	1.66	1.98	2.14	1.75	1.79	1.87
[Cl]/[Zn]	—	0.86	1.17	—	0.89	0.99
[C]/[Zn]	1.07	1.58	1.67	0.82	0.74	0.82

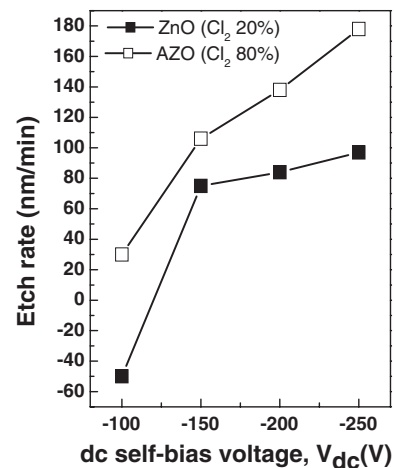


Fig. 5. Etch rates of AZO and ZnO as a function of the dc self-bias voltage (V_{dc}) at the ICP electrode power of 800 W, operating pressure of 15 mTorr, and etch time of 1 min.

increased monotonically with increasing V_{dc} . The results indicated that the ZnO and AZO etch process operates in the ion-assisted etching regime.^{11–13)}

Figure 6 shows the etch rates of AZO and ZnO as a function of top electrode power at the $\text{Cl}_2/(\text{Cl}_2 + \text{CH}_4)$ gas mixing ratio of 20 and 80%, respectively, and the V_{dc} of -250 V for 60 s. Etching was performed by varying the top electrode power from 500 to 800 W. The AZO and ZnO etch

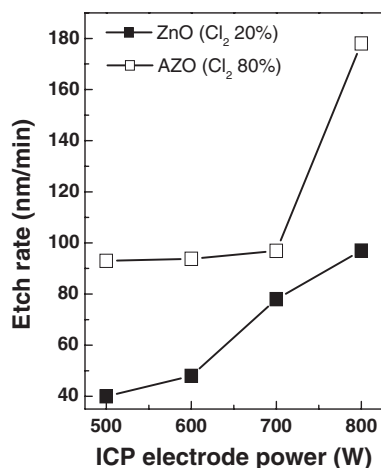


Fig. 6. Etch rates of AZO and ZnO as a function of the ICP electrode power at the V_{dc} of -250 V, operating pressure of 15 mTorr, and etch time of 1 min.

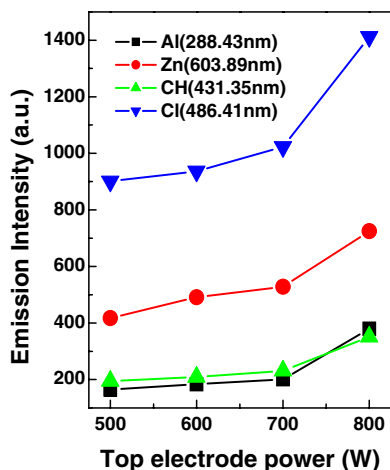


Fig. 7. (Color online) Optical emission intensities of the species during Cl_2 -based etching of AZO measured by OES as a function of ICP electrode power (etch conditions; dc self-bias voltage: -250 V, total gas flow rate: 100 sccm, Cl_2 flow ratios: 80%).

rates increased monotonically as the top electrode power was increased up to 700 W and then increased more rapidly at the top electrode power of 800 W. Increases in the Cl_x and CH_x radicals together with higher ion flux presumably contributed to the increase in the etch rate.

Figure 7 shows the optical emission intensities of the species in the plasma measured by OES under varying top electrode power. The Cl radical and CH radical emission intensities during AZO etching were increased with increasing top electrode power. The Al atom and Zn atom emission intensities were also increased, indicating the increase in the etch rates of AZO. The Cl and CH radical emission intensities increased more rapidly when the top electrode power was changed from 700 to 800 W, leading to the abrupt increase in the etch rate of AZO.

4. Conclusions

The etching characteristics of ZnO and AZO thin films were investigated by varying the process parameters in the Cl_2 - and BCl_3 -based ICPs. The etch rates of the ZnO and

AZO layers were higher in the Cl_2 -based chemistry than in the BCl_3 -based chemistry, probably because the more effective removal of oxygen atoms in the ZnO and AZO layers by chemical reaction of the oxygen atoms with the carbon atoms in the a-C:H layer increased their etch rates in the Cl_2 -based chemistry. The $\text{Cl}_2/(\text{Cl}_2 + \text{CH}_4)$ and $\text{BCl}_3/(\text{BCl}_3 + \text{CH}_4)$ flow ratios affected the etch rate variations of ZnO and AZO. For AZO etching, the effective removal of Al on the surface was very critical. Increasing the Cl_2 or BCl_3 flow ratio enhanced the removal of Al on the AZO surface due to formation of AlCl_x by-products, which are more volatile than Al-organic compounds and which in turn enhanced the AZO etch rate. For ZnO etching, the balance between Cl and CH_x radicals played an important role in determining the ZnO etch rate. Increasing the Cl_2 or BCl_3 flow ratio during ZnO etching reduced the Zn removal rate due to the decreased formation of $\text{Zn}(\text{CH}_x)_y$ by-products that resulted from the reduced CH_x radicals in the plasma.

Acknowledgments

This work was supported through the Center for Excellence program of the Korea Science and Engineering Foundation and the Ministry of Science and Technology (Grant No. R-11-2000-086-0000-0) and in part by the Second Stage of the Brain Korea21 Project in 2006 program through the Ministry of Education.

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