Effect of Low-Frequency Power on Etching Characteristics of 6H-SiC in C₄F₈/Ar Dual-Frequency Capacitively Coupled Plasma

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Abstract Dry etching of 6H silicon carbide (6H-SiC) wafers in a C₄F₈/Ar dual-frequency capacitively coupled plasma (DF-CCP) was investigated. Atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS) were used to measure the SiC surface structure and compositions, respectively. Optical emission spectroscopy (OES) was used to measure the relative concentration of F radicals in the plasma. It was found that the roughness of the etched SiC surface and the etching rate are directly related to the power of low-frequency (LF) source. At lower LF power, a smaller surface roughness and a lower etching rate are obtained due to weak bombardment of low energy ions on the SiC wafers. At higher LF power the etching rate can be efficiently increased, but the surface roughness increases too. Compared with other plasma dry etching methods, the DF-CCP can effectively inhibit C₆F₈ films’ deposition, and reduce surface residues.

Keywords: SiC, plasma etching, dual-frequency capacitively coupled plasma, X-ray photoelectron spectroscopy, optical emission spectroscopy

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1 Introduction

Because of its unique properties, such as large band gap, large breakdown field, excellent thermal conductivity, great hardness, and resistance to chemical attack and radiation, silicon carbide (SiC) is considered to be an excellent candidate for high-temperature, high-power and high-frequency electronic devices [1], micro-sensor and micro-actuator applications in micro-electromechanical systems (MEMS) [2], and applications in harsh environments [3]. The application of SiC to nano-devices requires precise etching of SiC and little surface residue after etching [4-6]. Unfortunately, the large Si-C bond energy makes SiC etching very difficult, therefore the powerful plasma-based dry etching becomes the main practical way to etch SiC in the fabrication of MEMS [7]. However, the surface residues resulting from dry etching can significantly affect the properties of materials, subsequent processing and device performance [8-9]. Therefore, it is very important to develop a new method to etch SiC with high etching rate, small surface roughness and little surface residue [7].

Dual-frequency capacitively coupled plasma (DF-CCP) is an important plasma etching tool which can be used for manufacturing ultra-large scale integrated (ULSI) circuits and other nano-materials. To achieve separate control of plasma density and energy of ions onto the wafer, two power sources with large discrepancy in frequency are usually used in the dual-frequency CCP reactor. The high-frequency (HF) source from 13.56 MHz to 500 MHz is used to control the plasma density, while the low-frequency (LF) source from 800 kHz to 2 MHz is used to adjust the energy of ions onto the wafer [10-14]. In our previous works on etching of SiCOH low-k films in CHF₃ DF-CCP, we have found that increasing the low-frequency power can effectively suppress the surface C₆F₈ residues [15,16]. However, the effect of low-frequency power on SiC etching is still unclear. Therefore, this work investigated the effect of power of low-frequency source on etching behavior of SiC in 60 MHz/2 MHz C₄F₈ DF-CCP.

2 Experimental setup

The n-type 6H-SiC wafers with the size of 5 mm×5 mm (from Tankblue Co. Ltd) were used as wafers. All the wafers were ultrasonically cleaned in HCl and NH₃ before being introduced into the vacuum chamber. After cleaning, wafers were immediately sent into the vacuum chamber. The etching of 6H-SiC wafers was carried out in a DF-CCP system. The plasma was produced between two symmetrical
parallel-plate stainless steel electrodes. The Comdel
CV500 RF generator (60 MHz, 300 W) was used as
the HF source and applied to the top electrode. The
HF power was obtained by subtracting the reflected
power from the incident power. The 6H-SiC wafers
were placed at the bottom electrode which was driven
by Comdel CX600 generator (2 MHz) in the power range
of 150 W to 300 W. The diameter of the two electrodes
was 200 mm and the gap between them was 50 mm.
The base pressure of the chamber was approximately
10⁻³ Pa. It is well known that F atom can react with
Si atom to produce a volatile species [17], therefore,
CₓFₘ/Arₓ mixed gas was chosen as the etching gas.
The flow rates of CₓFₘ and Arₓ were chosen as 10 sccm
and 0.5 sccm, respectively. The 0.5 sccm Arₓ was
used as the actinometry for the measurement of relative
concentration of F radicals. The working pressure was kept
at 40 Pa, and the etching time was 5 min.

The surface microstructure of etched 6H-SiC wafers
was analyzed by atomic force microscopy (AFM)
(Solver Pro SPM) with semi-contact operating mode.
All the measurements were performed on an area
of 500×500 nm². Chemical states of the pristine and
etched 6H-SiC wafers were identified by a Karotos
XSAN-800 X-ray photoelectron spectroscopy (XPS)
with an Al Kα irradiation (1486.6 eV) powered at
168 W with a resolution of 0.9 eV. The pressure in the
ion-pumped analysis chamber was 6×10⁻⁷ Pa during
data acquisition. The instrument was calibrated by Au
4f7/2 binding energy at 84.0 eV. Surface cleaning was
executed on the etched 6H-SiC in the XPS chamber
with 3 keV Ar⁺ ions (10 μA/cm², 5 min).

In order to analyze the effect of plasma on etching
behavior, relative concentration of F radicals was mea-
sured using optical emission spectroscopy (OES), which
was widely used in low pressure plasmas [18–21]. In our
experiment the optical emission was measured in the
wavelength range of 200–800 nm with a resolution of
0.05 nm to 0.13 nm using AvaSpec-2048 fiber optical
spectrometer equipped with a CCD detector. The op-
tical emission signal was collected via a fiber located
at approximately 2 mm above the surface of 6H-SiC
wafer. Fig. 1 shows the optical emission spectrum in
the wavelength range of 680–710 nm. A very sharp
and no Doppler broadening fluorine (F) line (703.8 nm)
can be observed. Because the Doppler broadening or-
originates from dissociative excitation, the result means
that the F line originates from electron impact excitation
rather than dissociative excitation [22]. Let nₓ be the
concentration of the free radical F and let Iₓ be the
optical emission intensity, the excitation emission from
the ground state F can be written as [23]

\[ Iₓ = αₓ nₓ, \tag{1} \]

where

\[ αₓ = k₃(λ) \int \frac{4πν²dνQₓs(ν)σₓ(ν)νf_e(ν)}{ν}, \tag{2} \]

Iₓ = \frac{C_F Ar-I_{Ar}}{I_{Ar}, \tag{3} \]

where

\[ C_F Ar = \frac{k₃(λF)Qₓs(ν)C_F}{k₃(λAr)Qₓs(ν)C_Ar}. \tag{4} \]

For the F (703.8 nm) and Ar (750.4 nm) emissions,
there are λF ≈ λAr, k₃(λF) ≈ k₃(λAr) and Qₓs(ν) ≈ Qₓs(ν) ≈ Qₓs(ν) ≈ Qₓs(ν). Thus, Eq. (4) can be written as
C_F Ar ≈ C_F Ar, which is related to the threshold behavior
of two cross sections. Because the excitation thresh-
old energy for F and Ar is nearly the same, 14.74 eV
and 13.47 eV, respectively, a good estimation of relative
concentration of F radicals nₓ can be directly obtained
by emissions intensity ratio of \( I_F \) vs. I_Ar.

3 Results and discussions

The variation of etching rate with the increase of LF
power is shown in Fig. 2. It can be seen that the etching
rate increases with the increase of LF power, and
reaches the maximum 152 mm/min at the LF power of
300 W. Fig. 3 shows the 3D AFM images of SiC wafers
before and after etching. The pristine SiC wafer has a
very smooth surface with the root mean square (RMS)
roughness of 0.33 nm, as shown in Fig. 3(a). After etch-
ing at the LF power of 150 W, the surface of the etched
SiC wafer becomes slightly rougher and the RMS rough-
ness increases to 0.4 nm. This result indicates that the
plasma etching at the LF power of 150 W causes lit-
tle roughness to the wafer. If the LF power is further
increased, the surfaces of etched SiC wafers become
rougner and rougher. Fig. 4 shows the change of surface
RMS roughness of etched SiC wafers with the increase of LF power. It can be seen that the RMS roughness increases with the increase of LF power. When the LF power increases to 300 W, the surface RMS roughness of the etched SiC wafer reaches 0.67 nm, which is twice that of the pristine wafer. Therefore, plasma etching at higher LF power can cause serious roughness to SiC wafers.

Fig. 2 Dependence of etching rate on low-frequency power

The dominant mechanisms related to etching behavior of 6H-SiC include physical and chemical mechanisms [17]. The physical mechanism is energetic ion bombardment. In the DF-CCP, the increase of LF power can lead to the increase of ion energy. The ion bombardment with high energy can cause more damage to SiC and result in a rougher surface. The chemical mechanism is the reaction between fluorine and silicon atoms to form volatile species [17]. In order to analyze chemical compositions of etched SiC, XPS measurements were carried out. Fig. 5 shows the C 1s core-level spectra and their Gaussian fitting peaks of (a) the pristine SiC wafer, and SiC wafers etched at the LF power of (b) 150 W to (e) 300 W, respectively. For the pristine SiC wafer, the C 1s core-level spectrum can be deconvoluted into two components. The peaks located at the binding energies of 282.6 eV and 284.4 eV are due to Si-C and C-C bonds, respectively [24]. For the SiC wafers etched at the LF power of 150 W to 300 W, C 1s core-level spectra can be fitted into three components. The peaks located at the binding energies of 282.6 eV, 284.4 eV and 288.0 eV correspond to Si-C, C-C and C-F bonds, respectively. However, compared with the C-C peak, the Si-C and C-F peaks are very small. For wafers (b) to (e), ratios of C-C to Si-C peak area are 7.2, 4.2, 5.6 and 6.6, respectively, while the ratios of C-C to C-F peak area for wafers (b) to (e) are 3.3, 2, 2.5 and 2.4, respectively. The small Si-C peak is due to the removal of Si atoms, while the weak C-F peak indicates small C$_x$F$_y$ residues on the surface of etched SiC wafers. In the C 1s core-level spectra, no C-F$_2$ or C-F$_3$ related bonds, which are located respectively at the binding energies of 289 eV [25] and 293 eV [26], are found. These results indicate that only very small C$_x$F$_y$ residues were left at the SiC surface after etching.

The etching characteristics of SiC wafers are mainly controlled by the fluorocarbon plasma chemistry. For the C$_4$F$_8$ gas, the dissociations by electron-neutral collisions are shown as follows [21,27],

\[ C_4F_8 + e \rightarrow F^- + C_4F_7, \quad \Delta H = 3.00 \text{ eV}, \quad (5) \]
\[ C_4F_7 + e \rightarrow C_2F_4 + C_2F_3 + e, \quad \Delta H = 2.42 \text{ eV}, \quad (6) \]
\[ C_4F_8 + e \rightarrow 2C_2F_4 + e, \quad \Delta H = 2.42 \text{ eV}, \quad (7) \]
\[ C_2F_4 + e \rightarrow 2CF_2 + e, \quad \Delta H = 3.06 \text{ eV}, \quad (8) \]
C$_2$F$_y$ films’ deposition on the etched materials can usually be observed in the fluorocarbon plasma etching, which can influence the etching characteristics [16]. But for SiC wafers etched by the DF-CCP, the XPS results show that very small C$_x$F$_y$ films deposit on the surface of SiC wafers. Therefore, the SiC etching with small C$_x$F$_y$ residues can be obtained by C$_4$F$_8$ DF-CCP.

The weak C$_x$F$_y$ residues on the surface of the SiC wafer may depend on the C$_x$F$_y$ radicals in the plasma and the energy of ions onto the SiC wafer. By OES measurement, the relative concentration of $F$ radicals is found to increase with the increase of LF power, as shown in Fig. 6. Because the F radicals are produced mainly by the dissociations of C$_4$F$_8$ gas, the increase of relative concentration of $F$ radicals means the increase of C$_4$F$_8$ dissociation. As a result, more C$_x$F$_y$ radicals can be produced in the plasma and deposit on the SiC wafers. However, the ion energy also depends on the LF power. LEE and HUANG’s results show that the increase of LF power can lead to the increase of ions’ energy and the broadening of ion energy distribution function (IEDF) to the high energy region [28,29]. Therefore, the ions’ bombardment onto the SiC surface can increase with the increase of LF power. As a result, more the C$_x$F$_y$ radicals sputtered, the less the C$_x$F$_y$ films left on the etched surface.

Therefore, the SiC etching involves the C$_x$F$_y$ films’ deposition, the ions’ energy and the relative concentration of F radicals. With the increase of LF power, both ions’ energy and relative concentration of F radicals increase. The increase of ions’ energy can lead to more sputtering of C$_x$F$_y$ films, and the increase of F radicals results in more removal of Si atoms in SiC by reacting with F radicals to form volatile products, thus the etching rate increases with the increase of LF power. Compared with other plasma dry etching methods, DF-CCP can effectively inhibit C$_x$F$_y$ films’ deposition. Therefore, the DF-CCP technique can be adopted as a useful tool for SiC etching with small surface residues.

4 Conclusions

In this work, the etching behavior of SiC wafers in the C$_4$F$_8$/Ar 60 MHz/2 MHz dual-frequency CCP was investigated. It is found that the etching rate and the surface roughness of etched SiC wafers are directly related with the low-frequency power. At lower LF power, the etched SiC surface is almost as smooth as the original one, and the etching rate is low. With the increase of LF power, the etching rate increases but the etched surface becomes rougher. It is due to the increase of energy of ions onto the etched SiC wafers. XPS and OES measurement results prove that the SiC etching depends on the C$_x$F$_y$ films’ deposition, the ions’ energy and the relative concentration of F radicals. With the increase of LF power, both ions’ energy and relative concentration of F radicals increase. The increase of ions’ energy can lead to more sputtering of C$_x$F$_y$ films, and the increase of F radicals results in more removal of Si atoms in SiC by reacting with F radicals to form volatile products, thus the etching rate increases with the increase of LF power. Compared with other plasma dry etching methods, DF-CCP can effectively inhibit C$_x$F$_y$ films’ deposition. Therefore, the DF-CCP technique can be adopted as a useful tool for SiC etching with small surface residues.

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