CHARACTERISATION OF A CRYOGENIC ETCHING SF₆/O₂
INDUCTIVELY COUPLED PLASMA DISCHARGE

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Abstract

The characterisation of an inductively coupled plasma discharge in SF₆/O₂ mixtures is of first interest for silicon etch process understanding and improvement. In the following, we will present electrical probe and actinometry measurements. Influence of discharge parameters such as the RF power, the gas fluxes, the pressure and the substrate bias on the electron temperature and density, the ion density and the relative atomic F and O concentrations will be discussed. We will also show measurements of the evacuated flux in different plasma conditions.

Keywords: Cryogenic plasma etching, SF₆/O₂ plasma characterisation, Actinometry, Electrical probe

1. Introduction

Due to the need of faster and highly anisotropic etch processes, the micro-electronic industry has induced highly dynamic researches in silicon etching, especially with inductively coupled plasma (ICP) reactor discharges. An etch process should present the following characteristics: a high etch rate (about several μm/mn), high anisotropy (side walls of the trench about 88-90°) and high selectivity (low etch rate of the mask), and should prevent contamination of the etched material. With this aim, a cryogenic process using SF₆/O₂ chemistry was developed [1] instead of the Bosch process, based on a fluorocarbon chemistry [2]. However, despite the large number of works, the etch mechanisms are not well known. As a consequence, understanding of mechanisms occurring in an ICP discharge during cryogenic etching of silicon is of first interest for improving the etch process (cf M. Boufnichel et al. paper « Deep etching of silicon in an ICP reactor using a cryogenic SF₆/O₂ chemistry process » in ISPC 2001 proceedings). The aim of this paper is to present results
concerning the characterisation by electric probe and actinometry of a SF₆/O₂ etching plasma in an industrial ICP reactor Alcatel 601E.

2. Experimental set-up

Experiments were performed in an Alcatel 601E Inductively Coupled Plasma (ICP) reactor [1]. Gases (SF₆, O₂ and Ar) were introduced in the source chamber by mass flow controllers. Gas fluxes were varied from 50 to 300 sccm for SF₆, and from 0 to 21 sccm for O₂. Ar flux was adapted in order to keep the ratio Ar/SF₆ constant and about 2.5%. Pressure ranged from 1 to 6.8 Pa and was controlled by a butterfly valve. RF (13.56 MHz) power was varied between 400 and 1400 W, and the substrate RF (13.56 MHz) bias was varied between 0 and −50 V. Each parameter was varied keeping the others constant, in the standard conditions as defined here: SF₆ flux 200 sccm, O₂ flux 13 sccm, RF power 1000 W, substrate bias −30 V, pressure 2.8 Pa, corresponding to the process conditions. In order to keep plasma conditions as stable as possible, substrates were SiO₂ wafers. The SiO₂ is the commonly used mask component, and the etch rate of the SiO₂ is very lower (about 300 times) than the one of silicon.

![Experimental set-up diagram](image)

Fig. 1: Experimental set-up

Plasma characterisation was performed by using a cylindrical electric probe (Sofie Digiprobe), for ion density and electron density and temperature measurements. The probe was introduced by a window in the diffusion chamber, 16 mm above the wafer. Electron temperature and density, ion density and plasma potential were determined from the I/V characteristic. The probe was moved parallel to the wafer surface in order to determine the spatial variations of plasma characteristics. Optical emission spectroscopy was used for determination of relative atomic fluorine and oxygen densities evolution by actinometry [3]. A Sofie Digisem optical spectrometer was used. By mean of a diaphragmed lens, a diaphragm located in the observation window and a beam dump located at the opposite of the observation window, a 3 mm diameter plasma tube in the diffusion chamber, parallel to the substrate surface, and 75 mm above it, was selected as the observed volume. The center of the chamber diameter was focussed on the entrance slit of the spectrometer. This set-up was conceived in order to avoid scattered light from the plasma source to reach the detector.

3. Results and discussion
Langmuir probe measurements

Characteristics of the plasma such as the plasma potential, the electron temperature and density and the positive ion density were determined in different plasma conditions. The electron temperature was measured to be about 2 - 4 eV, and is slightly influenced by the discharge parameters. $T_e$ increases from 2.3 to 3.5 eV when the substrate bias is varied between 0 and -50 V (see figure 2). This evolution is due to a variation of the plasma potential induced by the substrate bias voltage. An increase of the SF$_6$ flux from 50 to 300 sccm also induced an increase in the electron temperature from 2.6 to 3.2 eV. This result can be explained by an increased efficiency of electron attachment. $T_e$ is also slightly changed when the pressure is varied and exhibits a minimum at about 4 Pa (see fig. 5). We have also observed extrema on parameters such as the atomic fluorine and oxygen concentrations (see actinometry results), and on etch profiles (see M. Boufinichel et al. in ISPC 2001 proceedings) with pressure variation. When other parameters (power and O$_2$ flux) were varied, the electron temperature remained constant.

Figure 3 shows the influence of the pressure on the electron and ion densities. When pressure is increased up to 6 Pa, the ion and electron densities decrease from $2.2 \times 10^9$ to $8 \times 10^6$ cm$^{-3}$ and from $7 \times 10^7$ to $1 \times 10^7$ cm$^{-3}$ respectively. A part of the difference between the electron and ion densities can be due to the presence of negative ions in the discharge [4]. However, we have observed that, even in a pure argon discharge, the electron density was always lower than the ion density. An increase of the RF power (from 400 to 1400 W) induces an increase in the ion and electron densities (from $5 \times 10^7$ to $1.75 \times 10^8$ cm$^{-3}$ and from $5 \times 10^7$ to $4.5 \times 10^7$ cm$^{-3}$ respectively), due to an enhancement in the dissociation and ionisation processes. Measurements have shown that gas fluxes and substrate bias have no influence on the observed densities.

![Graph showing electron temperature and density with bias voltage and pressure](image)

Fig. 2: Evolution of the electron temperature $T_e$ with the bias voltage  
Fig. 3: Evolution of the ion and electron densities with the pressure

Actinometry

Evolution of relative densities of fluorine and oxygen atoms was determined by actinometry. A few argon flux was added to the discharge, and verified to not influence the
discharge characteristics. Then, the relative atomic concentrations were given by the ratio of fluorine and oxygen spectral lines intensities by the one of Arl 750.3 nm spectral line. Figure 4 shows the influence of the oxygen flux on the atomic F and O concentrations. We observe a quasi-linear variation of the O concentration with the O$_2$ flux. This indicates that the molecular oxygen dissociation is not at saturation. We also observed that the atomic fluorine concentration, deduced from different Fl spectral lines, increase with the O$_2$ flux. This could be due to chemical reactions in the discharge between SF$_x$ and O species such as the following: SF$_x$ + O → SOF$_{x-1}$ + F (1) [5]. As a consequence, such reactions increase the concentration of fluorine atoms.

Figure 5 shows the evolution of the F and O concentration with the pressure. We observe here that the concentrations of atomic species exhibit a maximum at around 4 Pa. It is noticeable that the electron temperature also shows a minimum at the same pressure. Moreover, we also observed a minimum on the trench characteristics at this pressure (cf M. Boufnichel et al. in these proceedings). This behaviour could be explained as follows. When the pressure is increased from 1 to 4 Pa, the residence time increases, improving the dissociation processes, and the densities of SF$_x$ and O$_2$ species also increase. As a consequence, atomic O and F concentrations increase. We have observed that the ion and electron densities decrease when the pressure increase (see fig. 3). Above 4 Pa, the increase of the residence time and of molecular species is not sufficient to counter-balance the decrease of the dissociation rate induced by the decrease of the electron density. Moreover, the ion density decreasing, the ion bombardment of the chamber walls decreases. This implies that the chamber walls can adsorb more reacting species such as F and O atoms, leading to a decrease of the concentration of these species. It is noticeable that oxygen concentration decreases faster than the one of fluorine. This could be explained by chemical reactions between SF$_x$, and O species.

![Graph showing relative density vs O$_2$ flux](image)

![Graph showing relative density vs O$_2$ flux and electron temperature vs pressure](image)

Fig. 4: Relative concentrations of fluorine and oxygen atoms vs O$_2$ flux

Fig. 5: Relative concentrations of F and O atoms and electron temperature vs pressure

Increasing the RF power up to 1000 W induces an increase of the atomic O and F concentrations, due to an increase of the electron density, thus to an increase of the dissociation rate. For a power higher than 1000 W, the O concentration continue to increase
while the F concentration saturates. This could be probably explained by a very high
dissociation rate of SF₆ molecules, implying that no more F atoms appear when the power
increase. Oxygen is probably relatively weakly dissociated. As a consequence, the O
centration continues to increase when the RF power is above 1000 W.

Varying the SF₆ flux from 50 to 300 seem appeared to have no influence on the atomic
F concentration, while the atomic O concentration decreases because the partial pressure of
molecular oxygen decreases. However, it appears that the O density decreases faster than the
partial pressure. This could be due to an increase of reactions (1) with the SF₆ flux. Actinometric results exhibit an obvious behaviour when the substrate bias is changed. O and
F concentrations decrease when the bias is increased. It is difficult to understand what kind of
influence the bias could have on O and F creation/recombination processes.

**Additional flux evacuated by the pumping group**

Additional flux was defined as the ratio of the measured evacuated flux when the
plasma is on, by the introduced flux, and measured by calibrating the position of the butterfly
valve with the flux, when plasma is off. The additional flux is not a measure of the
dissociation rate since the butterfly valve is located in front of the pumping group, relatively
far from the plasma source, where the dissociation mechanisms occur. However, the additional
flux is directly related to the dissociation rate.

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<th>Additional evacuated flux (%)</th>
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![Graph](image)

**Fig. 6 : Additional evacuated flux vs the O₂ flux**

**Fig. 7 : Additional evacuated flux vs the pressure**

Figure 6 shows the additional evacuated flux variation with the O₂ flux. It is noticeable
that it increases when the O₂ flux is increased. This observation is in good agreement with
preceding observation concerning the O₂ flux influence. Since O reacts easily with SF₆
species, it should be not responsible for the increase of evacuating flux. Moreover, reactions
(1) create F species, but no additional flux. If we consider that oxygen atoms react with the
chamber wall stronger than F atoms, reactions (1) limit the losses of species on chamber
walls. Thus, we could observe an increase of the evacuated flux.
Figure 7 shows the additional evacuated flux variation with the pressure. It appears to decrease faster at low pressures, and to saturate at high pressure. This can be due to the following causes. First, the electron density decreases when the pressure is increased, leading to a decrease of the dissociation rate. Second, the residence time increases and the mean free path decreases, improving the recombination reactions.

Lastly, an increase of the power induces a linear increase of the evacuated flux. It is noticeable that the electron density increases linearly with the RF power. As a consequence the dissociation rate, due to electron impact, increases linearly. The substrate bias appears to have no effect on the evacuated flux. If we consider that the dissociation essentially take place in the source, it is normal that the substrate bias has no effect on the dissociation since the source is located far (25 cm) from the substrate.

4. Conclusion

In conclusion, SF₆/O₂ ICP plasmas were characterised by means of Langmuir probe and actinometry diagnostics. Evolutions of parameters such as the electron temperature, the ion and electron densities and the atomic F and O concentrations were measured. Reactions between SF₆ species and O atoms appear to play an important role in dissociation processes.

References