Decomposition of SF$_6$ in an RF Plasma Environment

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ABSTRACT
Sulfur hexafluoride (SF$_6$)-contained gas is a common pollutant emitted during the etching process used in the semiconductor industry. This study demonstrated the application of radio-frequency (RF) plasma in the decomposition of SF$_6$. The decomposition fraction of SF$_6$, $\eta_{SF6} = (C_{in} - C_{out})/C_{in} \times 100\%$, and the mole fraction profile of the products were investigated as functions of input power and feed O$_2$/SF$_6$ ratio in an SiO$_2$ reactor. The species detected in both SF$_6$/Ar and SF$_6$/O$_2$/Ar RF plasmas were SiF$_4$, SO$_2$, F$_2$, SO$_2$F$_2$, SOF$_2$, SF$_{10}$, S$_2$OF$_{10}$, S$_2$O$_2$F$_{10}$ and SF$_4$. The results revealed that at 40 W, $\eta_{SF6}$ exceeded 99%, and the reaction products were almost all converted into stable compounds such as SiF$_4$, SO$_2$, and F$_2$ with or without the addition of oxygen. Sulfur oxyfluorides such as SO$_2$F$_2$, SOF$_2$, SOF$_4$, S$_2$OF$_{10}$ and S$_2$O$_2$F$_{10}$ were produced only below 40 W. The results of this work can be used to design a plasma/chemical system for online use in a series of a manufacturing process to treat SF$_6$-containing exhaust gases.

INTRODUCTION
Sulfur hexafluoride (SF$_6$) is commonly used as an etching/etching-aid gas in fabricating the submicrometer features of modern integrated circuits because it has a higher fluorine content than CF$_4$ but does not undergo polymerization.$^{1-3}$ However, the destruction of SF$_6$ has attracted much interest because of the important environmental issues and the toxicity of sulfur compounds.$^4$ Radio-frequency (RF) discharge plasma, which must be manipulated at low pressure, recently has become the most popular plasma technology used in the high-profit semiconductor industry, in both replicating patterns and depositing films. The discharge plasma can be manipulated at low substrate temperatures without changing its original properties and can replicate submicrometer patterns with anisotropic features.$^5$ However, in the presence of an RF discharge, SF$_6$, acting as an etching/etching-aid gas, can be decomposed into lower fluorides of sulfur and can generate hazardous byproducts, such as S$_2$F$_{10}$, SO$_2$F$_{2}$, SOF$_{2}$, SOF$_{4}$, and SF$_4$. Particularly toxic is S$_2$F$_{10}$, which has an LC$_{50}$ (concentration needed to kill 50% of a defined experimental animal population) of 0.1 mg/m$^3$$^{11}$. Because reducing or eliminating the toxicity of gaseous effluent from the RF discharge process is a serious concern, Breitbarth et al.$^{12}$ have proposed an oxygen-based RF discharge process in which a glass reactor is connected in series with a commercial RF parallel-plate reactor to decompose fluorocarbon waste gases into CF$_4$ and C$_2$F$_{10}$, which, in the presence of silica walls, are reacted to stable SiF$_4$. The SiF$_4$ then can be converted into nontoxic solid products with the aid of a CaO/Ca(OH)$_2$ absorber.

In this work, Ar and O$_2$/Ar RF plasmas were used to decompose SF$_6$. The decomposition fraction of SF$_6$, and the mole fraction profile of stable products in the effluent gas stream were determined. Optimal operating parameters, including input power and the feed O$_2$/SF$_6$ ratio, also were determined. Furthermore, an RF plasma treatment system connected to the semiconductor manufacturing process, which can both save energy and eliminate the toxic effluent, is discussed.
EXPERIMENTAL

Figure 1 presents the experimental apparatus used in this study. The flow rates of SF₆, O₂, and Ar were each monitored using a calibrated mass flow controller (Brooks 5850 E), and then mixed and introduced into a 4.14-cm i.d., 15-cm-long cylindrical glass reactor, at a fixed flow rate of 100 standard cm³/min (scm). The plasma discharge zone was wrapped with two external copper electrodes, which prevent the metal electrodes from participating in the reaction. These electrodes were coupled to a 13.56-MHz frequency RF generator (PFG 600 RF, Fritz Huttinger Elektronik Gmbh), which typically is used in commercial etching, with a matching network (Matchbox PFM). To prevent contamination, the pressure of the system was maintained below 10⁻³ Torr by a mechanical vacuum pump (PFEIFFER, DUO 065 DC) and a diffusion oil pump until the experiment was begun. Each design condition was achieved by measuring the concentration of reactants and products at least three times to ensure that the plasma reactions were in a steady state. The reactants and end products were first identified by gas chromatography/mass spectrometry (GC/MS, Varian Star/Saturn 2000) and a Fourier transform infrared (FTIR) spectrometer (Bio-Rad, Model FTS-7) and then were quantified using an online FTIR. Only neutral products were detected by IR spectroscopy and are presented here. Reactants and products both were calibrated by withdrawing unreacted gases directly through a sampling line connected to the FTIR. The mole fraction of the product species was determined by comparing with the response of the peak height of standard gas at the same wavenumber. However, the concentrations of SF₄ and S₂F₁₀ were estimated from data of Heise et al. and Wilmshurst and Bernstern. All unreacted F atoms were assumed to be converted into F₂, and the mole fraction of F₂ was determined using a mass balance. The plasma contact areas at various input powers were determined by multiplying the glow length by the circumference of the reactor. A feed concentration of 0.5% was selected to imitate the effluent concentration from industrial processes, because SF₆ concentrations as low as 1% in Ar can achieve high etch rates. Other parameters were as follows: The applied RF power was between 5 and 50 W, the total gas flow rate was 100 scm, the feed O₂/SF₆ ratio was between 0.0 and 2.0, and the operating pressure was 9 Torr. Feed O₂/SF₆ ratios of 0.0, 0.4, 1.0, 1.6, and 2.0 under input powers from 5 to 50 W were examined. However, because the distribution trends are similar among those feed O₂/SF₆ ratios, experimental results of feed O₂/SF₆ ratios of 0.4, 1.0, and 1.6 are not displayed.

RESULTS AND DISCUSSION

Decomposition of SF₆

A sensitivity analysis of an earlier model showed that the applied power was the most important parameter and positively affects the decomposition fraction of reactants in an RF plasma reactor. Figure 2 shows that when no oxygen was added to the reactor (feed O₂/SF₆ ratio = 0.0), η_SF₆ clearly increased from 37.79 to 95.68% because of an increase in power from 5 to 20 W. When the power exceeded 40 W, η_SF₆ exceeded 99%. When oxygen was introduced into the reactor (feed O₂/SF₆ ratio = 2.0), η_SF₆ was ~10% less than when no oxygen was added (from 28.61 to 86.09% as the power rose from 5 to 20 W). When the power exceeded 40 W, η_SF₆ exceeded 99%. When oxygen was introduced into the reactor (feed O₂/SF₆ ratio = 2.0), η_SF₆ was ~10% less than when no oxygen was added (from 28.61 to 86.09% as the power rose from 5 to 20 W). The addition of oxygen to the reactor reduced the electron density and, thus, reduced the dissociation of SF₆ and inhibited the production of free radicals. However, when the input power exceeded 40 W, η_SF₆ was not affected by the feed O₂/SF₆ ratio, leading to more than 99% decomposition.

Reaction Products

The major reaction products of the decomposition of SF₆ were SiF₄, SO₂, F₂, SO₂F₂, SOF₂, and SOF₄. The applied power was the most important factor that limited the production of all species. The mole fraction of SiF₄ increased from 0.18 to 0.23% when the input power was
varied from 5 to 20 W (Figure 3). Thus, more F radicals were produced, and the etchable wall surface also increased with the plasma contact area under higher input power (Figure 4), which was consistent with the assumption of Breitbarth et al.12 that the SiO₂ etch rate was proportional to the plasma power. However, as the power exceeded 40 W, ηSF₆ remained constant and resulted in the stable production of SiF₄. Neutral products such as SF₄, F atoms, and F radicals were observed and accounted for the high etch rate.7,18 The possible production mechanisms of SiF₄ are as follows:7,12

\[
4 \text{F} + \text{SiO}_2 \rightarrow \text{SiF}_4 + 2 \text{O} \quad (1)
\]

\[
\text{SF}_4 + \text{SiO}_2 \rightarrow \text{SiF}_4 + \text{SO}_2 \quad (2)
\]

\[
2 \text{SOF}_2 + \text{SiO}_2 \rightarrow \text{SiF}_4 + 2 \text{SO}_2 \quad (3)
\]

The etching of SiO₂ by F radicals, as in eq 1, might have dominated the process described here (see Figure 3), because the etch rate of Si was correlated with the intensity of F radical.19 The O₂ or O radicals did not compete with F in SiO₂ etching, which was consistent with the results of d’Agostino and Flamm.20

When no oxygen was added to the reactor, the mole fraction of SO₂ was under 0.05% at powers below 10 W (Figure 5). The fraction increased from 0.05 to 0.36% when power exceeded 10 W and gradually reached a steady state at 0.50% when the power reached 50 W. At this high power input, ηSF₆ exceeded 99% and led to the stable production of SO₂. The possible reaction processes are as follows:21

\[
\text{SF}_6 \rightarrow 4\text{F} \rightarrow \text{SF}_2 \rightarrow \text{F} \rightarrow \text{SF}_3 \rightarrow \text{F} \rightarrow \text{SF}_4 \rightarrow \text{F} \rightarrow \text{SF}_5 \quad (4)
\]

\[
\text{SF}_2 + \text{O} \rightarrow \text{SOF} + \text{F} \quad (5)
\]

\[
\text{SOF} + \text{O} \rightarrow \text{SO}_2 + \text{F} \quad (6)
\]

Oxygen radicals from the etching process can convert sulfur oxyfluorides into SO₂ most easily when no other oxygen sources are introduced. However, when another oxygen source was introduced into the system and reduced the electron density, the production rate of SO₂ was affected by the feed O₂/SF₆ ratio at powers under 20 W, leading to domination by sulfur oxyfluorides. When the power exceeded 20 W in the SF₆/O₂/Ar system, sulfur oxyfluorides were converted into SO₂. Those results also implied that, at high input power, the wall-etching process could release sufficient oxygen radicals to form products.

Interestingly, when oxygen was not a limiting factor, the production of SO₂F₂ was favored at ~20 W (Figure 6), which was consistent with the results of Ryan and Plumb, who used a microwave discharge.21
SO₂ + F → SO₂F  
SO₂F + F → SO₂F₂ (8)

However, the reverse reactions of eqs 7 and 8 could dominate the conversion of SO₂F₂ into SO₂ by endothermic processes, as follows:

SO₂F₂ = SO₂ + F₂  \Delta H° = + 462.6 kJ/mol (9)

The effluent gas temperature clearly increased when the input power exceeded 20 W (Figure 7). The gas-phase reactions were more likely to reach equilibrium at high temperatures and were favored for the endothermic reaction, because the operating pressure in this system was 1–2 orders higher than that of other RF discharges. However, when oxygen was not added to the system, the SiO₂ wall-etching reaction was the direct source of O radicals and easily led to the formation of SO₂ at low power inputs (see Figure 5). Figure 7 indicates that the temperature increased more steeply when no oxygen was added to the system than it did when oxygen was added. When the input power exceeded 40 W, the system favored the production of SO₂ and inhibited the formation of SO₂F₂.

Fluorine was assumed to be formed primarily in and immediately downstream of the discharge zone, as in Smolinsky and Flamm, as follows:

\[
2 F \xrightarrow{M} F₂  \quad M = \text{third body or wall} \quad (10)
\]

Production of F₂ (Figure 8) when no extra oxygen was added to the system followed a trend similar to that for SF₆ decomposition. When the feed O₂/SF₆ ratio was increased to 2.0, production of F₂ was affected by the competition of O₂ or O radicals with the F radicals to form sulfur oxyfluorides at powers below 20 W. As the input power increased, more F and O free radicals formed and reacted with the glass surface to form SiF₄, thereby hindering the formation of F₂. However, SF₆ decomposition also increased, promoting the generation of F₂.

The production of SOF₂ dominated at powers below 10 W (Figure 9). The SOF₂ concentration decreased nearly to 0 when the power exceeded 40 W and no oxygen was added to the reactor. When the feed O₂/SF₆ ratio was increased to 2.0, the concentration of SOF₂ increased slightly as the power was increased to 10 W and then declined gradually to almost 0 when the power exceeded 40 W.

When no oxygen was added to the system, the mole fraction of SOF₄ increased gradually from 0.024 to 0.07% as the power increased to 10 W (Figure 10), perhaps because...
a release of oxygen radicals from wall etching increased the probability of formation of SOF$_4$ by the following pathway:\textsuperscript{21}

\begin{equation}
\text{SF}_5 + O \rightarrow \text{SOF}_4 + F \tag{11}
\end{equation}

As the power was increased, the production of SO$_2$ dominated that of SOF$_4$. Increasing the feed O$_2$/SF$_6$ ratio to 2.0 reduced the electron density and slowly increased the mole fraction of SOF$_4$. Formation of SO$_2$ and F$_2$ was suppressed simultaneously.

The minor decomposition products were S$_2$F$_{10}$, S$_2$OF$_{10}$, S$_2$O$_2$F$_{10}$, and SF$_4$ (Figures 11-14). Oxygen radicals can inhibit the formation of S$_2$F$_{10}$ by eq 11.\textsuperscript{3,22} The production of S$_2$OF$_{10}$ and S$_2$O$_2$F$_{10}$ was enhanced as the feed O$_2$/SF$_6$ ratio increased from 0.0 to 2.0 and at over 10 W. The possible reaction mechanisms are as follows:\textsuperscript{22}

\begin{align*}
\text{SF}_5 + O + M & \rightarrow \text{SF}_5\text{O} \tag{12} \\
\text{SF}_5\text{O} + \text{SF}_5 + M & \rightarrow \text{S}_2\text{OF}_{10} \tag{13} \\
2 \text{SF}_5\text{O} + M & \rightarrow \text{S}_2\text{O}_2\text{F}_{10} + M \tag{14}
\end{align*}

The formation of S$_2$O$_2$F$_{10}$ reached its maximum value at an input power of 20 W. However, the mole fraction of S$_2$OF$_{10}$ reached its maximum value at an input power of 30 W and decreased steeply almost to 0 when the power exceeded 40 W. The SF$_4$ was almost undetectable in this work. However, its concentration decreased with both increased power input and increased feed O$_2$/SF$_6$ ratio.

**Suggested Treatment Process**

An online treatment process connected an RF plasma system to a commercial RF dry-etching process, without
influencing low-pressure operation, because gaseous effluent almost could be converted into stable products such as $\text{SiF}_4$, $\text{SO}_2$, and $\text{F}_2$ when power exceeded 40 W. However, the high toxicity of these stable compounds is such that the simple plasma conversion of these products seems not to suffice in the treatment of $\text{SF}_6$-containing exhaust gases. An absorber consisting of primary CaO can convert $\text{F}_2$ and $\text{SiF}_4$ into $\text{CaF}_2$, as follows:12

$$\text{F}_2 + \text{CaO} \rightarrow \text{CaF}_2 + 1/2 \text{O}_2 \quad (15)$$

$$\text{SiF}_4 + 2 \text{CaO} \rightarrow 2 \text{CaF}_2 + \text{SiO}_2 \quad (16)$$

Fluorspar (CaF$_2$), known as an F-containing mineral, is employed primarily in high-temperature smelting and refining processes to produce metals and alloys.23 Rees et al.23 showed that F ions were barely biologically available because of the low aqueous solubility of CaF$_2$. Soda ash, the most common and efficient reagent in flue gas desulfurization, can be used, in a regenerative method, to treat the $\text{SO}_2$ effluent gas as follows:24

$$\text{Na}_2\text{CO}_3 + \text{SO}_2 \rightarrow \text{Na}_2\text{SO}_3 + \text{CO}_2 \quad (17)$$

With all units connected in series, an RF plasma process may be an alternative method for treating $\text{SF}_6$-containing gases.

**CONCLUSIONS**

The reaction products in the effluent stream of $\text{SF}_6/\text{Ar}$ and $\text{SF}_6/\text{O}_2/\text{Ar}$ plasma systems included $\text{SiF}_4$, $\text{SO}_2$, $\text{F}_2$, $\text{SO}_2\text{F}_2$, $\text{SOF}_2$, $\text{S}_2\text{OF}_{10}$, $\text{S}_2\text{OF}_{11}$, $\text{S}_2\text{OF}_{12}$, and $\text{SF}_4$. Experimental results showed that $\eta_{\text{SF}_6}$ depended strongly on input power. As the power increased from 5 to 50 W, $\eta_{\text{SF}_6}$ increased from 28.61 to 99.79% and from 37.79 to 99.87% with and without added oxygen, respectively. Operating parameters were suggested as follows: The $\text{SF}_6$ feed concentration was 0.5%, the applied RF power was 40 W, the feed O$_2$/SF$_6$ ratio was 0.0, the total gas flow rate was 100 sccm, and the operating pressure was 9 Torr. The $\text{SF}_6$ and sulfur oxyfluorides were converted into stable compounds such as $\text{SiF}_4$, $\text{SO}_2$, and $\text{F}_2$ by etching of the SiO$_2$ wall. Sulfur oxyfluorides such as $\text{SO}_2\text{F}_2$, $\text{SOF}_2$, $\text{S}_2\text{OF}_{10}$, and $\text{S}_2\text{OF}_{11}$ were formed only at powers below 40 W. When the power exceeded 40 W, sulfur oxyfluorides were converted into stable products. The feed O$_2$/SF$_6$ ratio did not affect the distribution of products. However, when the power was lower than 40 W, adding oxygen resulted in a lower electron density and suppressed the formation of $\text{SiF}_4$, $\text{SO}_2$, and $\text{F}_2$. Increasing the feed O$_2$/SF$_6$ ratio promoted the formation of sulfur oxyfluorides such as $\text{SO}_2\text{F}_2$, $\text{SOF}_2$, $\text{S}_2\text{OF}_{10}$, and $\text{S}_2\text{OF}_{11}$. The results of this work can be used to design a process for treating $\text{SF}_6$-containing gases emitted in semiconductor manufacturing processes.

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