



Microwave plasma removal of sulphur hexafluoride

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ABSTRACT

Sulphur hexafluoride (SF_6) gas is a common pollutant emitted during the plasma etching of thin films and plasma cleaning chemical vapor deposition (CVD) production processes used in the semiconductor industry. In this paper a method using microwave (2.45 GHz frequency) plasmas sustained at atmospheric pressure for the abatement of SF_6 is investigated experimentally for various gas mixture constituents and operating conditions, with respect to its ability to decompose SF_6 to less harmful molecules. The destruction and removal efficiencies (DRE) of plasma abatement of SF_6 at concentrations between 1.7 and 5% in nitrogen in the presence of water vapor were studied as a function of the total gas flow rate and microwave power. Water vapor proved to be an effective source of free radical species that reacts with the radicals and ions resulting from SF_6 fragmentation in the plasma and also, it proved to reduce the process by-products. It was measured that ~25% of the initial SF_6 is converted to SO_2 . Destruction and removal efficiencies of SF_6 up to 99.9% have been achieved.

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1. Introduction

Air pollutants may be considered primary – emitted directly into the air – or secondary pollutants that are formed in the air by chemical and/or photochemical reactions on primary pollutants. The formation of secondary pollutants, such as tropospheric ozone and secondary aerosols, from primary pollutants such as SO_2 , NO_x , NH_3 and volatile organic compounds (VOCs) is strongly dependent on climate and atmospheric compositions [1].

Amongst air pollutants, an important category consists of greenhouse gases, gases that affect the planet's heat balance. These gases can contribute to the greenhouse effect both directly and indirectly. Direct effects occur when the gas itself is a greenhouse gas like water vapor, carbon dioxide (CO_2), methane (CH_4), nitrous oxide (N_2O) and ozone (O_3).

Indirect radiative forcing occurs when chemical transformations of the original gas produce other greenhouse gases, when a gas influences the atmospheric lifetimes of other gases, and/or when a gas affects atmospheric processes that alter the radiative balance of the earth. Very powerful greenhouse gases that are not naturally occurring include perfluorocompounds (hereafter PFC), e.g. SF_6 , CF_4 , CHF_3 , NF_3 , C_2F_6 . At present, the largest use of PFC (a few hundred tonnes per year) is by the semiconductor industry [2]. Amongst them SF_6 is the most potent greenhouse gas with the highest global warming potential ($\text{GWP}_{100} = 23,900$) and an

atmospheric lifetime of 3200 years [3] making SF_6 highly dangerous if exhausted to the atmosphere. SF_6 emissions vary depending on a number of factors: gas used, type/brand of equipment used, company-specific process parameters, number of SF_6 -using steps in a production process and whether abatement equipment has been implemented. Generally, process tools consume from 15 to 60% of influent SF_6 depending on the chemical used and the process application (etch or CVD) [4,5]. It follows that the waste gases will contain 40–85% of the input amounts of SF_6 so to further reduce emission levels, process optimization, alternative chemicals, capture/recycling and effluent abatement are all being considered. Also, before the exhaust gas is vented to the atmosphere, an abatement device is often provided to treat the exhaust gas. The abatement device converts the more hazardous components of the exhaust gas into species that can be readily removed from the exhaust gas, for example by conventional scrubbing and/or can be safely exhausted to the atmosphere.

This paper is concerned with the effective decomposition of SF_6 using a high pressure microwave (2.45 GHz frequency) plasma induced and sustained within a resonant cavity.

2. Considerations of gas abatement using atmospheric pressure plasmas

At the present time, the only field-proven technique for treating SF_6 -containing exhaust streams is thermal, either by direct thermal oxidation or catalytic oxidation. In thermal treatment the object is to transfer heat to the exhaust stream with either electrical resistance losses or burning fuel providing the original source

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of heat. This method is indirect and can be somewhat fuel or electricity intensive. It is within this context that, over the past 10 years or more, atmospheric pressure plasmas have been increasingly promoted as a technology for a number of applications in the area of pollution abatement and it is intended to improve on some of the shortcomings of thermal systems. Atmospheric pressure plasmas have a big advantage over low-pressure plasma due to the fact that atmospheric plasmas do not require a containment vessel, hence allowing continuous processing on a large scale.

It is by now well established that optimal mixtures of some particular reactants can be produced in a weakly ionized medium only if the electron temperature is much greater than the ion and gas temperatures. Previous research on SO_2/NO_x removal from flue gas showed that a minimum electron temperature $T_e \approx 10\text{--}15\text{ eV}$ was required to produce significant yields of active species such as OH radicals and monoatomic nitrogen and oxygen while if the gas temperature (T_g) was allowed to increase towards equilibrium values, recombination would reduce the concentration of the active species more rapidly than the desired reactions could take place [6,7]. It appears that only in the non-equilibrium state with $T_e \gg T_g$ can the desired reactions proceed at an appreciable rate. Furthermore, many chemical reactions rates can be greatly increased if the reactants are in excited vibrational states that would not be populated to an appreciable extent in an equilibrium plasma [8]. Therefore, there is a growing interest in using non-equilibrium plasmas for different chemical applications. In addition, for gas cleaning applications, such plasmas do not require additional fuel material as per combustion technologies, since they generate high energy electrons capable of forming reactive free radicals that facilitate the decomposition process, whilst maintaining relatively modest ambient temperatures.

Physically, non-thermal equilibrium in atmospheric pressure discharges is a relatively marginal solution. In practice there are three excitation concepts to generate such discharges. To date, environmental applications have been using mainly corona or dielectric barrier discharge (DBD) systems. However, these concern essentially low concentrations (10–100 ppm) in medium to high air flows (10–1000 m^3/h). Electron densities in corona or DBD plasmas hardly exceed 10^{11} cm^{-3} in streamers and 10^9 cm^{-3} in homogeneous volume [9–11]. In practice, this is totally insufficient to achieve appropriate PFC conversion rates in the typical conditions that prevail at the exhaust of a primary vacuum pump, namely 0.3–0.5% relative concentrations in 20–50 L/min nitrogen or air.

Microwave (MW) plasma differs significantly from other plasmas, exhibiting many interesting properties. For example, the electron density and temperature is higher in MW plasma than in radio-frequency or dc plasmas, so its reactivity is expected to be higher. The reason of this being the fact that at high frequencies, only light electrons can follow the oscillation of the electric field. This way they gain energy and collide mainly by elastic collisions with both the molecules to be abated and the carrier gas. At atmospheric pressure, microwave plasmas exhibit homogeneous densities of $10^{12}\text{--}10^{15}\text{ cm}^{-3}$ and therefore seem much more attractive for effluent abatement in the aforementioned flow and concentration conditions [9]. In addition, a large number of inelastic electron-neutral collisions result in very efficient dissociation at relatively high concentrations of the gas to be abated. Such inelastic collisions proceed continuously preventing the reformation of the initial molecules. Microwave plasmas can be excited inside resonant cavities, within waveguide microwave circuits or by means of surface wave field applicators [12,13].

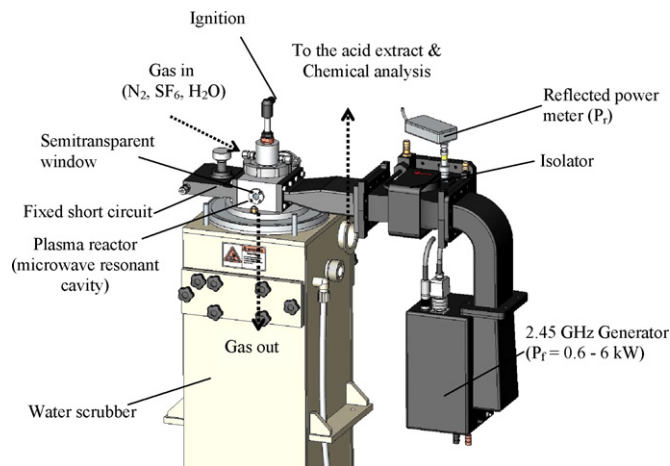


Fig. 1. Schematic of the experimental system.

3. Experimental

3.1. Microwave and plasma set-up

The microwave system used to initiate and create the discharge is shown in Fig. 1. It consists of a microwave generator (power supply and magnetron) with the power output adjustable from 0.6 to 6 kW at a frequency of 2.45 GHz, a water cooled isolator (maximum rating of 6 kW) with crystal detector for reflected power measuring, a rectangular waveguide, a single-mode microwave cavity and a microwave short circuit designed with a stub tuner to compensate for any changes in impedance loading, thus optimizing power transfer from the source to the gas to be abated.

The plasma source in this investigation is based on a single-mode microwave rectangular cavity with an internal volume $V = 120\text{ cm}^3$ within which the active zone of the plasma discharge is enclosed [14–16]. The resonant cavity enables highly efficient coupling of microwave radiation to the plasma, due to careful design such that a standing wave is established at a precise point in the enclosure. The cavity is made from a section of a standard WR340 waveguide that at 2.45 GHz frequency operates in the fundamental mode, yielding TE_{10} mode for the cavity. The electric field distribution is represented in Fig. 2(1).

A very accurate texture of all surfaces and the choice of stainless steel as material for the plasma reactor is not only a requisite to minimize wall losses and to maximize the quality factor Q of the single-mode microwave rectangular cavity but also to ensure sufficient reliability of the plasma reactor exposed to the highly energetic and corrosive plasma in fluorinated nitrogen and water vapor.

Microwaves are fed into a WR340 waveguide which is tapered to raise the electric field in front of the coupling section of the cavity. The discharge is operated at atmospheric pressure; the breakdown is initiated and maintained by the field enhancement inside the resonator cavity from power levels as low as 600 W. Microwaves with a frequency of 2.45 GHz are fed into the plasma reactor resulting in a high field concentration in the middle of the cavity—Fig. 2(2). In this region, the plasma is ignited and heated. Several kilowatts can be injected into the plasma. The breakdown and subsequent behaviour of the plasma is observed visually through the semitransparent window of the resonant cavity as well as by recording the value of the reflected power.

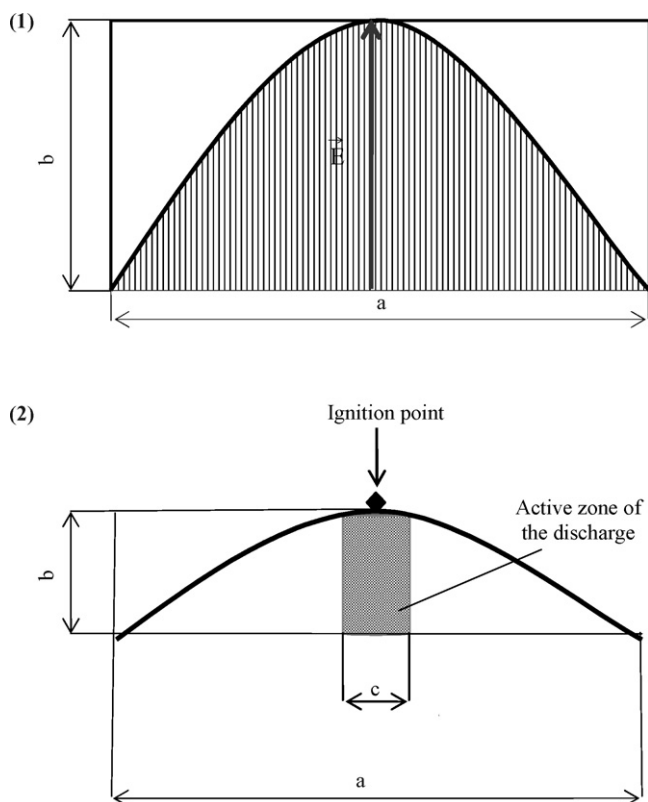


Fig. 2. (1) Electric field, \vec{E} , distribution and intensity in TE₁₀ mode microwave waveguide ($a = 86.4$ mm, $b = 43.2$ mm). (2) Dimensions of the plasma reactor and the active zone of the discharge $a = 86.4$ mm, $b = 20$ mm, $c = 10$ mm. Active zone of the discharge is the area over which the interaction between the electromagnetic field and the discharge takes place.

3.2. Chemical analysis

The analyses of the gas mixtures before and after reaction were monitored on-line by FT-IR analysis. FT-IR spectra were collected with an MKS MultiGas 2030 on-line spectrometer, fitted with KBr optics, a multi-pass sample cell of 5.1 m path length (operating at 150 °C) and a liquid-nitrogen cooled MCT (Cadmium Mercury Telluride) detector. Absorption spectra were collected between 500 and 5000 cm^{-1} at 0.5 cm^{-1} resolution. To improve the signal-to-noise ratio, averages of 32 spectra were stored once every 18 s. Gas was sampled through the sample cell at 3 L/min flow rate. Under these flow conditions, the gas pressure in the sample cell is 1 atmosphere. Spectra were analyzed and displayed using On-Line's quantitative spectral library as well as in house calibrations.

The destruction and removal efficiencies of SF₆ (DRE) were calculated:

$$\text{DRE (\%)} = \frac{C_{\text{SF}_6\text{i}} - C_{\text{SF}_6\text{f}}}{C_{\text{SF}_6\text{i}}} \times 100 \quad (1)$$

where $C_{\text{SF}_6\text{i}}$ is the initial concentration (ppm) of SF₆ and $C_{\text{SF}_6\text{f}}$ is its final concentration after the plasma abatement and water scrubbing—Fig. 3.

4. Results and discussion

A microwave plasma source can be realized in many different ways. However, there are some general considerations that are common to all: the plasma results from a gas discharge sustained by an electromagnetic field and the conditions for its reactivity are essentially determined by the charged particle and energy loss

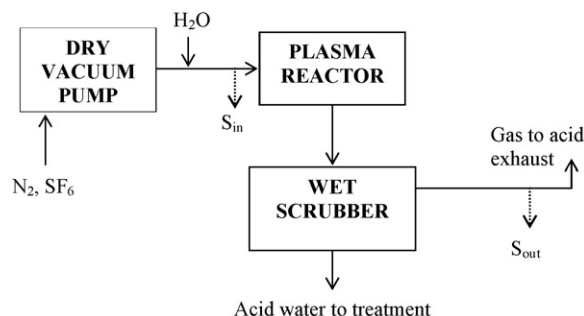
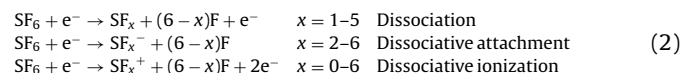


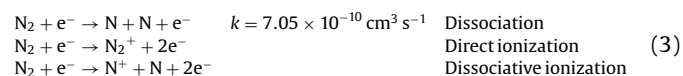
Fig. 3. Diagram of the experimental system with sampling ports S_{in} —inlet sampling port; S_{out} —outlet sampling port.

mechanisms. The charged particles within the discharge are accelerated by the electric component of the electromagnetic field. At microwave frequencies the ions, because of their large mass, remain practically unaffected by the field and can be considered motionless [13].

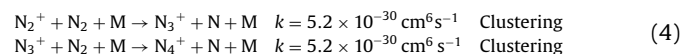
In the case of a gaseous mixture of SF₆ and N₂, the following reactions tend to dominate the behaviour of the ionized gas at lower temperature with higher electron temperature. SF₆ can be directly ionized, attached or dissociated by electron-impact processes. Because of its electronegative nature, SF₆ is able to form negative ions either by direct attachment or dissociative attachment—Eq. (2),



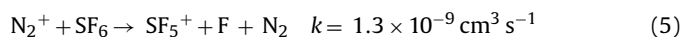
where all the reaction rates depend on the electron temperature (T_e). According to [17], dissociative attachment governs SF₆ decomposition when $T_e < 5.5$ eV, while direct dissociation predominates when $T_e > 5.5$ eV. However, since the concentration of SF₆ is much lower than that of N₂, electron-impact processes are less likely to occur than the N₂ processes. The primary electron-impact processes will then be the dissociation and ionization of nitrogen, Eq. (3).



Since present processes occur in high gas pressure, three body-reactions take place and yield, Eq. (4) [18].



SF₆ can also be dissociated through ion-molecule reactions [18,19], Eq. (5).



When using H₂O_(g) as an additive, electron-impact dissociation produces O, H, and OH radicals. In comparison with other methods described in literature [17–20], water is more economic and provides simultaneously both the H and O containing species needed in the abatement process [20–23]. A schematic of all possible reac-

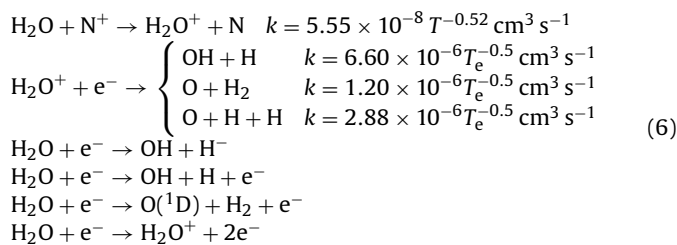
Table 1
Plasma pyrohydrolysis of sulphur hexafluoride (SF₆)

Run	Inlet		Outlet (gaseous by-products)							
	N ₂ (L/min)	C _{SF₆} ^a (%)	SF ₆ C _{SF₆} (ppm)	DRE (%)	SO ₂ (ppm)	SOF ₂ (ppm)	SO ₂ F ₂ (ppm)	NO (ppm)	NO ₂ (ppm)	N ₂ O (ppm)
1	60	1.7	14	99.9	3244	<1	<1	2149	28	58
2		3.3	25	99.9	8539	<1	<1	3077	79	178
3	80	1.7	32	99.8	1908	<1	<1	1625	20	12
4		3.3	68	99.8	6720	<1	<1	2535	69	90
5	100	1.7	223	98.9	4718	8.5	<1	1317	33	49
6		3.3	673	97.7	5070	110	4.8	1551	58	80

Destruction and removal efficiencies (DRE) and by-products distribution dependence on nitrogen flow and SF₆ initial concentration. P_r=6 kW; H₂O:SF₆ = 5:1.

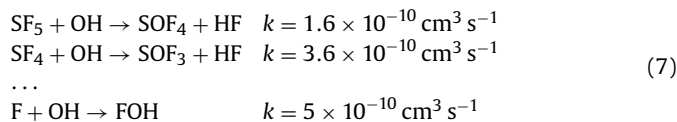
^a Calculated values using SF₆ and N₂ initial flow rates (measured with mass flow controllers).

tions is described below, Eq. (6).

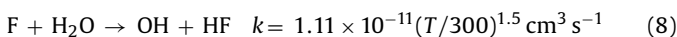


where T_e is measured in eV and T in degrees K.

H and O are precursors for SF₆ remediation; however, OH can also remediate SF₆ by both oxidizing SF_x ($x=1-5$) and preventing its reassociation with F to make SF₆ [24]–Eq. (7).



Water can also directly react with free fluorine (F) atoms that might otherwise recombine to form SF₆ [23].



This reaction also generates OH, which is readily available for further abatement via a chain reaction.

When considering the kinetics of the plasma abatement process, it is noteworthy to mention that the chemistry in continuous wave microwave plasmas is different to the chemistry in other electric discharges leading to different by-products. As a rule, microwave discharges yield small and simple molecules that can be easily scrubbed. This feature results from the large number of electrons colliding with heavy particles and the continuous action of these collisions throughout the abatement cycle which, first, contribute to break the pollutant molecule into atoms and small molecular fragments and, second, limit the size of any by-product [25].

Considering the high density of the microwave plasma (high intensity and frequency of the electric field) within the microwave reactor, the proposed reaction mechanism for SF₆ conversion in the presence of water is pretty simple yielding only SO₃ and HF–Eq. (9).



4.1. Effect of SF₆ initial concentration and residence time

The sole reagent for SF₆ destruction used in this investigation is water; the reaction stoichiometry in Eq. (9) indicates that 1 molecule of SF₆ requires 3 molecules of water for complete conversion to SO₃ and HF. In reality, this ratio was determined to be much higher and must be kept close to 5:1 (H₂O:SF₆). However, the

FT-IR results of exhaust gas analysis show a mixture of by-products including SO₂–Table 1.

As shown in Table 1, the destruction of the SF₆ molecule depends mainly on the microwave power density and the gas flow, i.e. the residence time. The residence time of molecules within the microwave plasma reactor decreases when the gas flow rate is increased resulting in decreased DRE, e.g. 99.9% DRE at 60 L/min (run 2) and 97.7% DRE at 100 L/min (run 6). When keeping the gas pressure, microwave power and reagent ratio constant but increasing the concentration of SF₆ molecules, the DRE value can stay constant (runs 1–4) or decrease (runs 5 and 6). A decrease in DRE is mainly due to more SF₆ molecules being rebuilt because the absolute number of their non-oxidized fragments in the plasma increases. An increased number of fragments of the initial molecule can also lead to a higher number of by-products such as SOF₂.

Based on the results in Table 1, the following secondary reactions (10) and (11) are suggested within the wet scrubber. In these experiments, the maximum quantity of SO₂ formed was ~25% of the initial SF₆.

Secondary reactions (downstream plasma):



According to [26] the SO₃ loss is second order in the water concentration, is independent of pressure and has a strong negative temperature dependence. The yield of sulphuric acid is 1.0 ± 0.5 per SO₃ consumed. These observations are consistent with the rapid association of SO₃ and H₂O to form the adduct H₂OSO₃ which reacts with water to produce sulphuric acid.

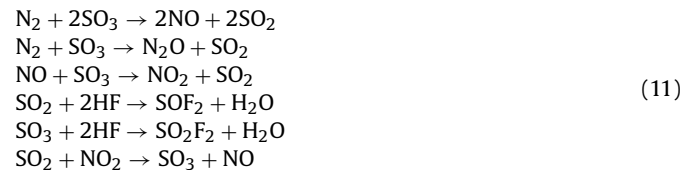
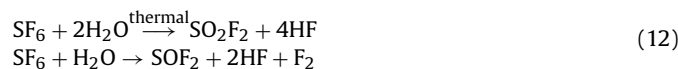


Table 1 also shows that, under constant microwave power, the DRE values for the two SF₆ concentrations considered (runs 1 and 2, and runs 3 and 4) are the same as a function of the N₂ flow rate. The slight decrease of DRE in runs 5 and 6 as a function of N₂ flow rate implies that the DRE is directly dependent on the time available for the oxidation of SF₆ fragments, i.e. decreased residence time of the gas to be abated within the microwave plasma, i.e. 0.11, 0.08 and 0.07 s corresponding to runs 1, 3 and 5 and 0.1, 0.075 and 0.06 s corresponding to runs 2, 4 and 6, respectively. The formation of small quantities of SO₂F₂ and SOF₂ as by-products of the oxidation process is attributed to the thermal oxidation of SF₆ [27] consistent with a lower gas temperature downstream plasma due to increased

N₂ flow—Eq. (12).



4.2. Effect of microwave power

The dominant feature of plasmas at atmospheric pressure is the low degree of ionization and the very high collision rates, especially of electrons with neutral species. At the electron densities n_e typically attained, the plasma behaves as a very lossy dielectric or rather poor conductor. The microwave field induces currents which are dissipated resistively. The dependence between DRE_{SF_6} on microwave forward power is shown in Fig. 4. DRE_{SF_6} increases as the microwave forward power is increased from 2 to 6 kW.

A set of experiments was performed to determine the optimum microwave forward power (P_f) for an efficient abatement of SF_6 , i.e. $\text{DRE}_{\text{SF}_6} \geq 99\%$. As shown in Fig. 4, increasing the microwave forward power (P_f) greatly increases the SF_6 removal speed and efficiency. However, as the total gas flow changes the microwave power absorbed by the plasma changes as well. To understand how the energy transferred from the plasma to the gas phase influences the removal efficiency of SF_6 , the value of absorbed microwave power was used to estimate the energy required to obtain $\text{DRE}_{\text{SF}_6} \geq 99\%$. The absorbed microwave power by the plasma, P_{plasma} , was cal-

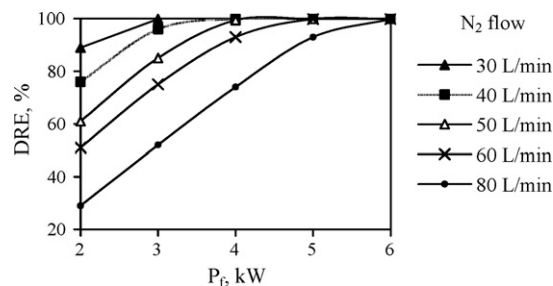


Fig. 4. Destruction and removal efficiencies of SF_6 (DRE) dependence on the microwave forward power (P_f) and N_2 flow rate. $\text{SF}_6 = 2$ L/min, $\text{H}_2\text{O}:\text{SF}_6 = 5:1$.

culated as the difference between the total applied microwave forward power (P_f) and the reflected power (P_r) measured for each set of experiments—Table 2. In the absence of any electron temperature measurements and electron density calculations, P_{plasma} was used to calculate the optimum energy required for the abatement process. This estimation is considered by the authors appropriate to evaluate the power dissipated within the plasma as in the absence of the plasma the value of reflected power is equal to that of the forward power emitted from the microwave generator, i.e. no power dissipation within the resonant structure in the absence of the plasma. The results in Table 1 and Fig. 4 indicate that the

Table 2

Required level of plasma energy (E_{plasma}) transferred to a mixture of $\text{N}_2 + \text{SF}_6 + \text{H}_2\text{O}$ for $\text{DRE}_{\text{SF}_6} \geq 99\%$

N ₂ flow (L/min)	80	60	50	40	30
Total gas flow N ₂ + H ₂ O + SF ₆ (L/min)	92	72	62	52	42
SF ₆ concentration (% vol.)	2.2	2.8	3.2	3.9	4.7
Microwave forward power, P_f (W)	6000	5000	4000	4000	3000
Microwave reflected power, P_r (W)	600	700	400	700	300
Absorbed microwave power, P_{plasma} $P_{\text{plasma}} = P_f - P_r$ (W)	5400	4300	3600	3300	2700
Energy density required for $\text{DRE}_{\text{SF}_6} \geq 99\%$, E_{plasma} (W ³ min/L)	58.7	59.7	58.1	63.5	64.3

$\text{SF}_6 = 2$ L/min, $\text{H}_2\text{O}:\text{SF}_6 = 5:1$.

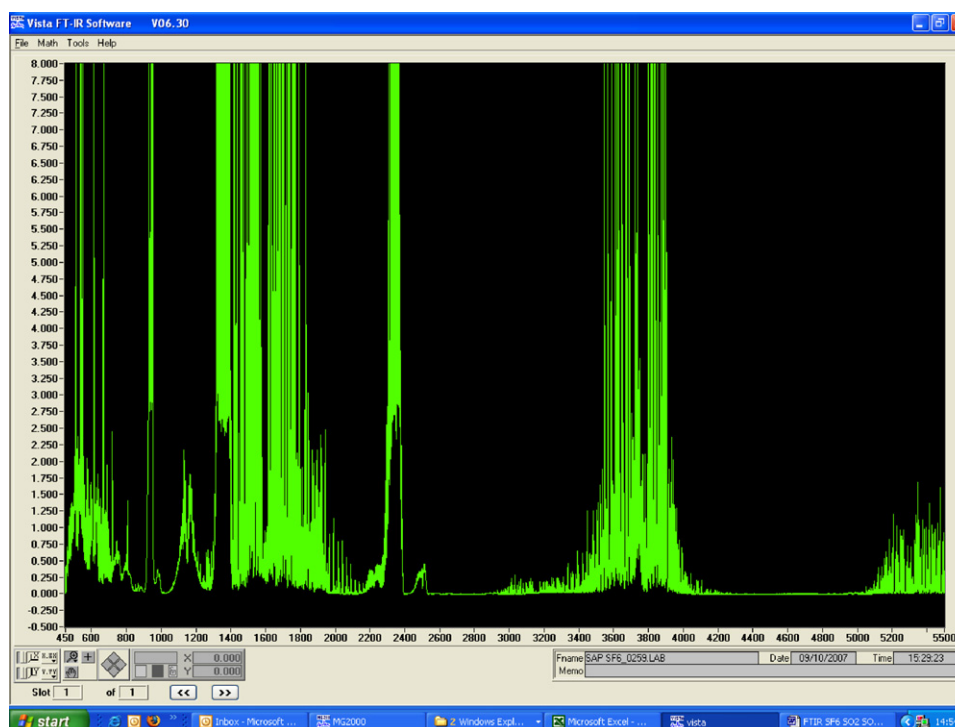


Fig. 5. FT-IR recorded spectrum (absorbance vs. wave number, cm^{-1}) for run 6, Table 1.

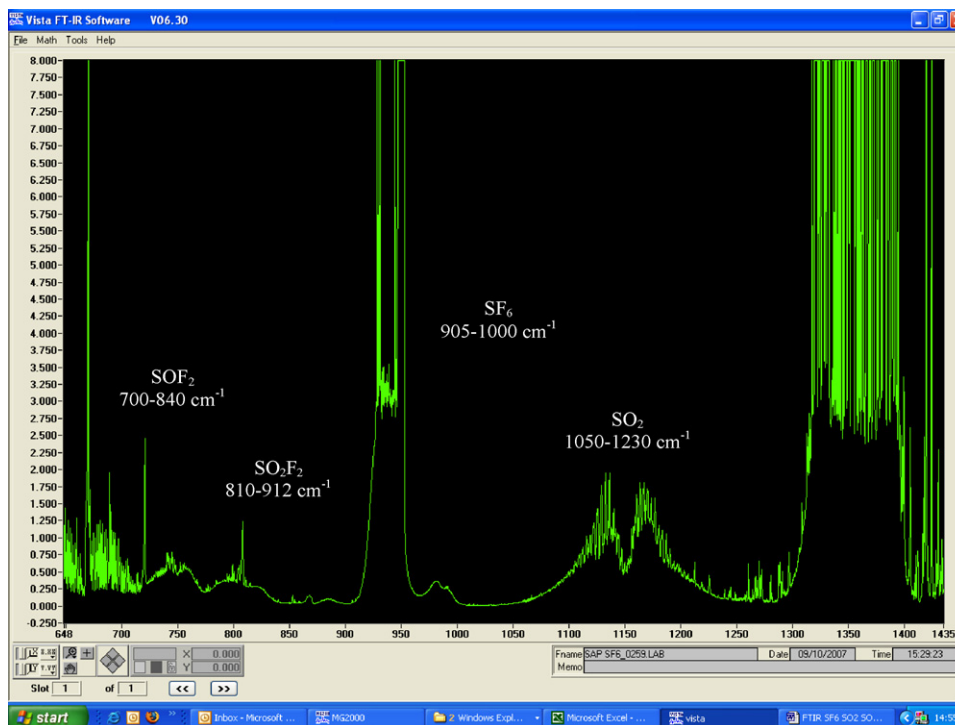


Fig. 6. FT-IR spectrum (run 6, Table 1) showing SF₆ and its abatement by-products.

decomposition rate of SF₆ is dependent on the total flow rate and energy and less dependent on the SF₆ concentration.

The result in Table 2 shows that the optimum energy transferred from the plasma to the gas to be abated is 58 W min/L. Energy levels higher than this value do not seem to improve the abatement efficiency. These results are in agreement with theoretical results presented in literature. According to [28], at low n_e , the microwave field is unaffected by the presence of plasma and the power absorbed is approximately proportional to the number of electrons present and their collision rate. As the n_e increases, the absorbed power starts to reduce the electric field in the cavity and the induced currents prevent full penetration of this field into the plasma by skin effect (for a definition of skin effect see Appendix A). At very high n_e , the skin effect is strong enough to exclude field from the plasma bulk and heating is restricted to a surface layer leading to decreased efficiency of the abatement.

4.3. Gaseous by-products

It was measured that up to 25% of the initial SF₆ is converted to SO₂. A clear mechanism of SO₂ formation was not established and we shall further extend these results with more studies focused on by-products formation and changes as the reaction proceeds. As SO₂ is not a desirable by-product, the main scrubbing after the plasma should be reconsidered, e.g. dry scrubbing, dosing of the wet scrubber with peroxides or potassium permanganate, hot oxygen addition, excess of fresh water. Small quantities of SO₂F₂ and SOF₂ were also detected when the total flow rate and the SF₆ concentration through the plasma reactor increased—Figs. 5 and 6.

5. Conclusion

Methods of controlling the waste volatiles from the semiconductor industry may not yet appear to compare in sophistication to the chemistry of wafer processing. In part, this is a consequence over

the years of a lower research effort by the industry and its suppliers on pollution control than on wafer production, for the profits of the industry are generated from sales of processed wafers. In addition, semiconductor manufactures deal with great changes every year: shorter device product lifecycles, rapid technology changes and market volatility. Therefore, the abatement method must have the flexibility to react quickly and effectively to market and technology changes.

The potential of MW sustained plasma technologies for controlling the waste volatiles from the semiconductor industry has been addressed in this paper. The scope of this paper is to demonstrate the ability of microwave plasma treatment to induce a variety of physical and chemical phenomena, which can play a significant role in the development of new and existing technologies in the greenhouse gas abatement.

An investigation has been conducted for removal of SF₆ by 2.45 GHz microwave plasma discharges. The conversion of process gases was performed at atmospheric pressure in N₂ dilution gas and excess water vapor as additive. Water vapor proved to be an effective source of free radical species that reacts with the radicals and ions resulting from SF₆ fragmentation in the plasma and also, it proved to reduce the process by-products. By-products such as FO_xN_y, FO_z, COF_t were not observed and SF₆ conversion by-products were relatively simple molecules. This is attributed to the physical properties of the microwave discharge with high electron density and relatively low energy.

The results show that the removal efficiency of the overall system decreased with increasing total gas flow, i.e. on the purge gas (N₂), which dominated the composition of the effluent stream. Removal efficiency increased with microwave total power level.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jhazmat.2008.07.112.

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