



Intermediate species in a 2.45 GHz microwave plasma sustained in an argon–tetramethylsilane gas mixture

J.L. Jauberteau*, I. Jauberteau, J. Aubreton

SPCTS, UMR 6638 CNRS, UER des Sciences, 123 av A. Thomas, 87060 Limoges, France

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Abstract

A microwave discharge (2.45 GHz) sustained in an argon–tetramethylsilane (Ar–SiMe₄) gas mixture is investigated by means of mass spectrometry in order to study H₂SiMe⁺ and HSiMe⁺ ions. These species are supposed to proceed via SiMe₃⁺ ion rearrangement process. We show that in our experiments they mainly results from the electron impact on desorbed species from the reactor wall and not from rearrangement processes. We have measured an ionisation threshold for H₂SiMe⁺ and HSiMe⁺ ranges between 10 and 11.5 eV and 10 and 10.5 eV, respectively. These values are lower than the value given in the literature (13.6 and 14 eV) when the ions proceed via SiMe₃⁺ ion rearrangement process. In the case of SiMe₂⁺, the threshold ionisation measured is also lower than the value given in the literature when this ion is produced by the dissociation ionisation of SiMe₄. This ion is also probably produced by ionisation of desorbed species. Such effect of desorbed species in our experiments could be explained by the reactor design. The length between the discharge and the mass spectrometer is rather long in this reactor configuration and the species are deposited on the reactor wall. These results are discussed and reaction schemes are given.

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Keywords: Tetramethylsilane; Plasma; Desorbed species; Ionisation; Threshold ionisation

1. Introduction

Plasma discharge containing organosilicon compound are widely employed for thin films deposition applications because of the specific properties of their polymers. The tetramethylsilane (SiMe₄ where Me is the methyl group) is the simplest organosilicon compound used as a precursor in plasma assisted chemical vapour deposition of silicon nitride or silicon carbide films [1–4]. The control of film deposition process

need a good knowledge of the plasma composition. The modelling of the complex chemical system implicated in the discharge require a good understanding of the different elementary reactions.

This work is devoted to the study of particular reactive processes involved in an argon–tetramethylsilane microwave plasma deposition process. We study H₂SiMe⁺ and HSiMe⁺ ions which are supposed to be produced in an ion rearrangement process. The contribution of these ions to the total ionisation is important (more than 20%), it is just after the SiMe₃⁺ contribution one [5]. They are expected to play a great role in these reactive plasma.

* Corresponding author. Tel.: +33-5-55-45-75-03;

fax: +33-5-55-45-72-11.

E-mail address: jauberteau-ijl@unilim.fr (J.L. Jauberteau).

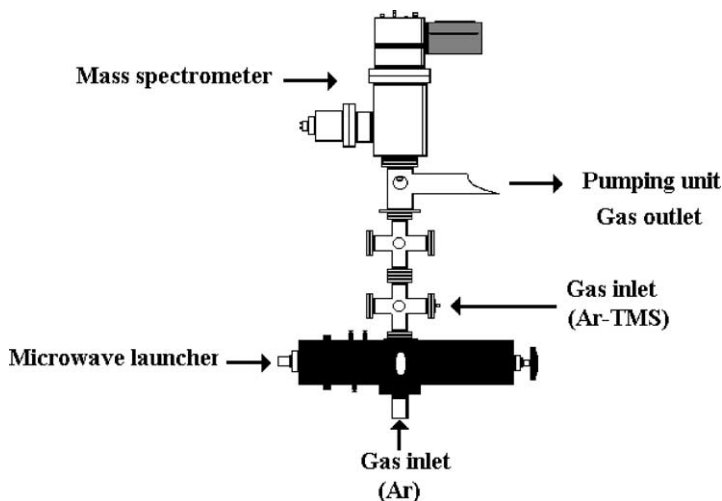


Fig. 1. The experimental set-up.

2. Experimental set-up

The experimental set-up is schematically shown in Fig. 1. It consists of a pure argon microwave discharge working at 2.45 GHz in a quartz tube (outer diameter 19 mm, inner diameter 16 mm). In order to maintain a constant temperature, the outside of the quartz tube is refreshed by means of an air cooling. The microwave discharge is sustained at an argon pressure equal to 66 Pa and a microwave power fixed equal to 200 W. A stainless steel tube with an inner diameter equal to 50 mm is located just above the discharge and the gas flow in this tube is kept constant and equal to 20 m/s by means of a Roots blower pumping unit. The tetramethylsilane is a liquid so the vapour (saturated vapour pressure at 293 K) is mixed to argon carrier gas in a small stainless steel vessel fixed on the gas line. Then, the gas mixture is injected in the reactor at 1 cm above the exit of the quartz tube containing the argon discharge. In this part of the quartz tube the glow discharge is produced and consequently the gas mixture is injected in the discharge.

The gas composition is analysed by means of a quadrupole mass spectrometer analyser (QMG 421 Balzers) equipped with a secondary electron multiplier amplifier. Since, the mass spectrometer cannot

operate at a pressure larger than 10^{-3} Pa within the quadrupole, a two-stages differential pumping unit is required. It consists of two pumping units composed with a turbomolecular pump and a dry primary pump, one of them is connected to the intermediate chamber between the flowing tube and the quadrupole chamber and the second is connected to the quadrupole chamber. During the gas sampling, the pressure is typically of 66 Pa or more in the reactor and in the flowing tube and of 10^{-3} Pa in the intermediate chamber (first pumping stage) and 10^{-5} Pa in the quadrupole chamber (second stage).

The mass spectrometer sample hole is located at 20 cm above the argon–tetramethylsilane injector exit. The diameter of the sample hole is 100 μm and the diameter of the hole between the intermediate chamber and the quadrupole chamber is 500 μm .

3. Results and discussion

When the discharge is working and the argon–tetramethylsilane is injected in the reactor, several species are detected in the reactor. They are identified using the ionisation threshold method [6–8]. This technique relies on the fact that most molecule take more energy

Table 1

<i>m/z</i>	Ions	Reactant	Ionisation threshold (eV)	Detection threshold (eV)
1	H ⁺	H	13.56	>15
2	H ₂ ⁺	H ₂	15.426	15–16
14	CH ₂ ⁺	CH ₂	10.50	10–11
	CH ₂ ⁺	CH ₄	15.19 (0.02)	
	N ⁺	N ₂	24.32	
	N ⁺	N	14.53	
15	CH ₃ ⁺	CH ₃	9.84 (0.002)	10–12
	CH ₃ ⁺	CH ₄	15.19 (0.02)	
	NH ⁺	NH	12.8	
16	CH ₄ ⁺	CH ₄	12.71 (0.01)	11–12
	NH ₂ ⁺	NH ₂	11.22	
	O ⁺	O ₂	18.98 (0.05)	
17	NH ₃ ⁺	NH ₃	10.166	Not detected
	OH ⁺	OH	18.19 (0.1)	
	OH ⁺	H ₂ O	13.18 (0.1)	
18	H ₂ O ⁺	H ₂ O	12.62	12–13
28	Si ⁺	Si	8.151	10–12.5
	N ₂ ⁺	N ₂	15.58	
	C ₂ H ₄ ⁺	C ₂ H ₄	10.51	
	C ₂ H ₄ ⁺	C ₂ H ₆	12.08	
	CO ⁺	CO	14.013	
	H ₂ O ⁺	H ₂ O	12.62	
29	SiH ⁺	SiH	8.01 (0.01)	<10
		SiH ₄	15.3 (0.3)	
30	SiH ₂ ⁺	SiH ₄	11.90 (0.02)	11–12
	NO ⁺	NO	15.01	
	NO ⁺	NO ₂	12.34	
	C ₂ H ₆ ⁺	C ₂ H ₆	11.521 (0.007)	
	N ₂ H ₂ ⁺	N ₂ H ₂	9.85 (0.1)	
31	SiH ₃ ⁺	SiH ₄	12.30 (0.03)	Not detected
32	O ₂ ⁺	O ₂	12.059	12–13
	SiH ₄ ⁺	SiH ₄	11.66	
	N ₂ H ₄ ⁺	N ₂ H ₄	8.36	
34	H ₂ O ₂ ⁺	H ₂ O ₂	10.92 (0.052)	10
40	C ₃ H ₄ ⁺	C ₃ H ₄	9.64	15–16
	CO ₂ ⁺	CO ₂	13.77	
	Ar ⁺	Ar	15.75	
42	C ₃ H ₆ ⁺	C ₃ H ₆	9.74	10–11
	SiCH ₂ ⁺	HSiMe ₃	10.6 (0.3)	
43	SiMe ⁺	H ₃ SiMe	14.05 (0.05)	10–11
	SiMe ⁺	H ₂ SiMe ₂	14.0 (0.15)	
	SiMe ⁺	HSiMe ₃	12.4 (0.3)	
	SiMe ⁺	SiMe ₄	17.1 (0.4)	
	C ₃ H ₇ ⁺	C ₃ H ₇	8.10 (0.05)	
44	SiO ⁺	SiO	11.67	10–10.5
	CO ₂ ⁺	CO ₂	13.76	
	N ₂ O ⁺	N ₂ O	12.89	
	C ₃ H ₈ ⁺	C ₃ H ₈	10.95	
	HSiMe ⁺	H ₃ SiMe	11.45 (0.05)	
	HSiMe ⁺	H ₂ SiMe ₂	10.85 (0.05)	
	HSiMe ⁺	HSiMe ₃	11.0 (0.3)	
	HSiMe ⁺	SiMe ₄	16.3	
45	H ₂ SiMe ⁺	H ₃ SiMe	11.8 (0.05)	10–11.5

Table 1 (Continued)

m/z	Ions	Reactant	Ionisation threshold (eV)	Detection threshold (eV)
	H ₂ SiMe ⁺	H ₂ SiMe ₂	11.51 (0.05)	
	H ₂ SiMe ⁺	HSiMe ₃	12.8 (0.5)	
	H ₂ SiMe ⁺	SiMe ₄	13.81 (0.02)	
	H ₂ SiMe ⁺	(SiMe ₃) ⁺ *	12.81	
	N ₃ H ₃ ⁺	N ₃ H ₃	9.6 (0.1)	
	C ₂ H ₅ O ⁺	C ₂ H ₅ O	9.11	
46	NO ₂ ⁺	NO ₂	9.75 (0.01)	9–10
56	Si ₂ ⁺	Si ₂	7.3	10–11
57	SiC ₂ H ₅ ⁺	Si(C ₂ H ₅) ₄	19.4	10–11
58	C ₄ H ₁₀ ⁺	<i>n</i> -C ₄ H ₁₀	10.5	10–11
	NMe ₃ ⁺	NMe ₃	9.8	
	NC ₃ H ₈ ⁺	(C ₂ H ₅) ₂ NH	9.55	
	SiMe ₂ ⁺	H ₂ SiMe ₂	10.71 (0.05)	
	SiMe ₂ ⁺	HSiMe ₃	10.3 (0.2)	
	SiMe ₂ ⁺	SiMe ₄	13.9 (0.3)	
69	C ₅ H ₉ ⁺	C ₅ H ₉	7.79 (0.03)	10–11
70	Si ₂ N ⁺	Si ₂ N	9.4 (0.3)	10–12
	CSiMe ₂ ⁺			
71	CHSiMe ₂ ⁺			10
72	CH ₂ SiMe ₂ ⁺			10
73	SiMe ₃ ⁺	HSiMe ₃	10.52 (0.05)	10–11
	SiMe ₃ ⁺	SiMe ₄	10.09 (0.02)	
74	HSiMe ₃ ⁺	HSiMe ₃	9.8 (0.3)	10
80	Ar ₂ ⁺	Ar + Ar*	14.71 (0.009)	14–15
88	SiMe ₄ ⁺	SiMe ₄	9.86 (0.02)	9.5–10

The peaks detected for various m/z values (first column). The ionisation threshold values measured (last column) compared to the ionisation threshold value given in the literature (fourth column) for different species (second column).

to accomplish dissociation ionisation than direct ionisation. Ions produced via a direct ionisation process is observed at a threshold ionisation value lower than the same ion produced via a dissociation ionisation process. Consequently, the ionisation process can be deduced from the appearance potential measured if we compare this value to data given in the literature and corresponding to different ionisation processes already studied. In Table 1, we have compiled the different m/z values observed in the mass spectrum (first column of the table). We report on the appearance energy (ionisation threshold values measured given in the last column) and compare these values to the ionisation threshold value given in the literature (fourth column) for different ions (second column) which are produced by collision between electrons with various reactants (third column). The main peaks are observed at $m/z = 88, 73, 58, 45, 44, 43, 40, 28, 15, 2$ and 1. According to Table 1, they can be ascribed to SiMe₄⁺,

SiMe₃⁺, SiMe₂⁺, H₂SiMe⁺, HSiMe⁺, SiMe⁺, Ar⁺, C₂H₄⁺, CH₃⁺, H₂⁺, H⁺, respectively. Figs. 2 and 3 display the signal intensity measured for some main species vs. the electron energy when the discharge is on and off, respectively. For most of these species (excepted $m/z = 45$) the signal measured when the discharge is on is slightly lower than the signal measured when the discharge is off. This behaviour can be explained assuming that these species are largely dissociated in the discharge at 200 W, producing simpler radicals or molecules [6]. In both cases the ionisation threshold remains the same for all the species.

It is worth noting that the distance between the mass spectrometer sample hole and the discharge exit is 20 cm and the tetramethylsilane injector is placed 1 cm above the discharge exit. This reactor configuration is different from the reactor configurations previously used in reference [6], where we have shown that the reactor design has a large effect on the tetramethylsilane

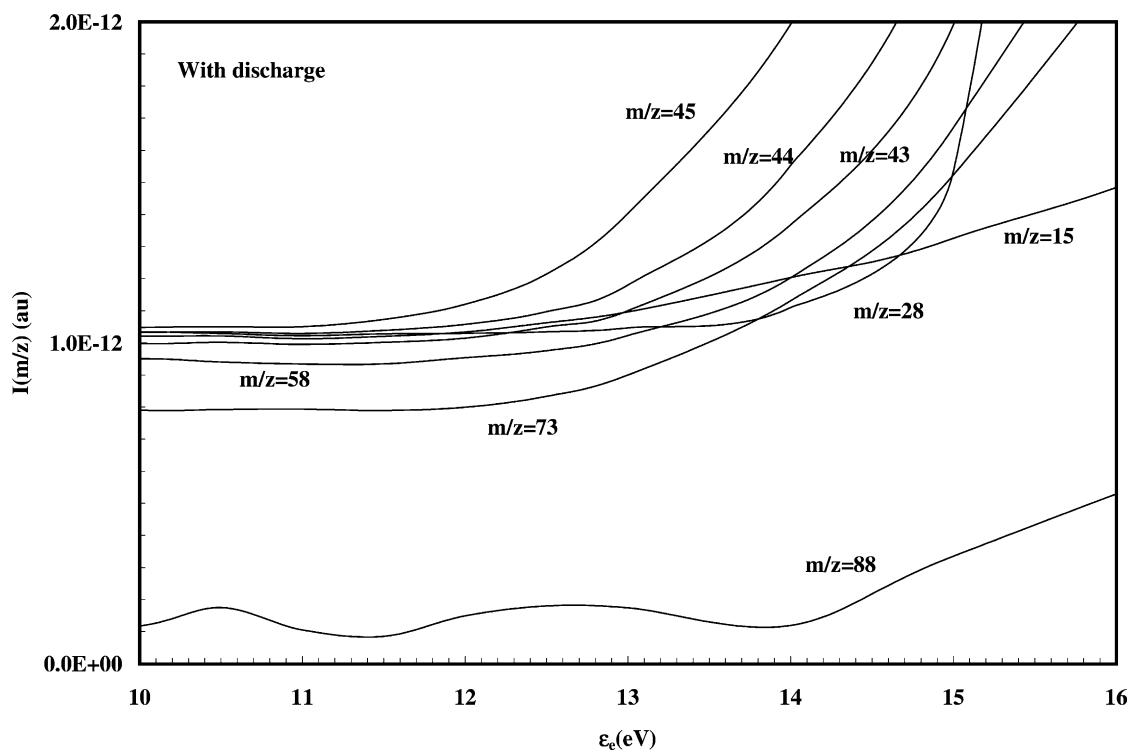


Fig. 2. The change in the mass spectrometer signal intensity measured vs. the electron energy for some main species when the discharge is on.

dissociation yield and consequently on the plasma chemistry. So the results presented in this paper are partly different from results given in reference [6]. This is the case of the relative intensity measured for $m/z = 58, 73$ or 43 . It can be noted that for $m/z = 45$ and 44 , the curves exhibit a shoulder at low electron energies, which is only observed when the discharge is off. This behaviour shows that different mechanisms and probably different reactants are implicated to produce these ions when the discharge is on and off. As it is related elsewhere [6], most of the species produced results of Si–C or C–H bond breaking. Because Si–C and C–H binding energies are equal to 76 and 99 kcal/mol, respectively, the tetramethylsilane dissociation proceed mainly by Si–C bond breaking producing heavy radicals ($\text{SiMe}_{x<4}$) at low microwave power (lower than 40 W). At a microwave power larger than 40 W, the C–H bond breaking mechanism becomes efficient producing smaller radicals (CH_x and SiH_x).

The following part of this paper is focused on the study of H_2SiMe^+ and HSiMe^+ ions, these species are expected to be produced via other mechanisms that simple Si–C or C–H bond breaking. According to Litzow and Spalding [11], the formation of so called “hydride ions” in MMe_4 ($\text{M} = \text{Si, Ge, Sn,}$) spectra, results of H rearrangements involving the elimination of CH_2 radical from one methyl group or C_2H_4 from two methyl groups. These reactions are supposed to be metastable supported in some case. Monohydride ions (Me_2MH^+ , MeMH^+ , MH^+) are observed for all tetramethyl compounds. Me_3MH^+ ion is observed for $\text{M} = \text{Ge, Sn, Pb}$ and other ions (MH_2^+ , NH_3^+ , MeMH_2^+) are observed in the spectra of the silicon and germanium compounds.

According to Distefano [10], the most abundant ions observed in the photoionisation mass spectrum of SiMe_4 , are SiMe_4^+ , SiMe_3^+ , H_2SiMe^+ and the threshold ionisation values ranges between 9.69 and

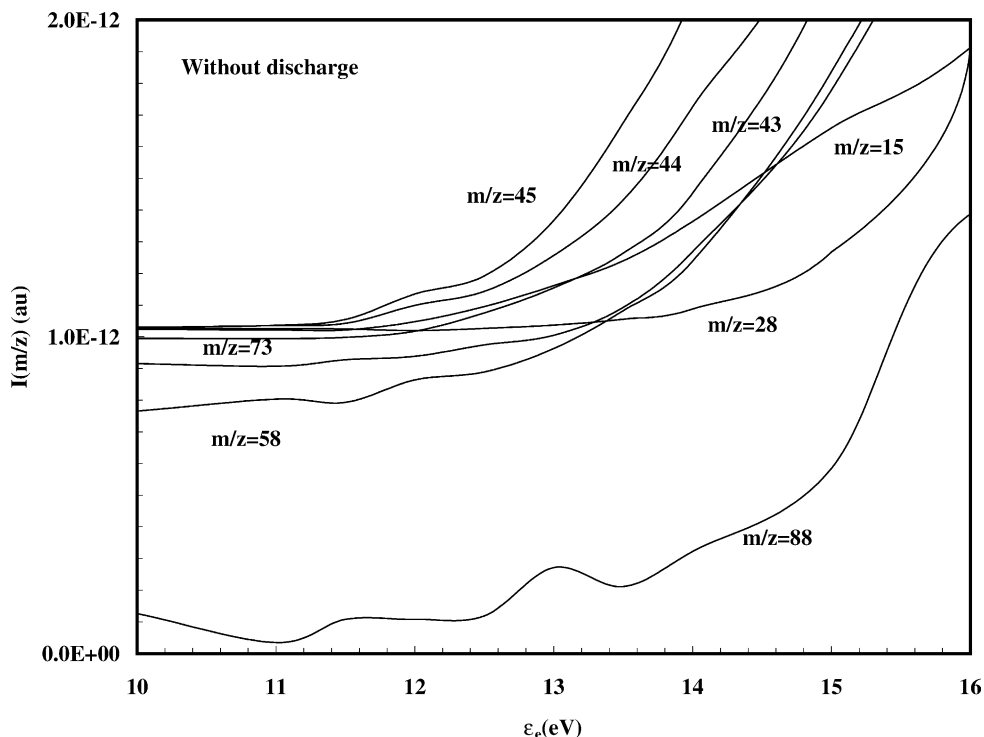
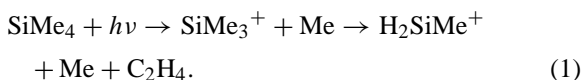


Fig. 3. The change in the mass spectrometer signal intensity measured vs. the electron energy for some main species when the discharge is off.

9.86, 9.90 and 10.09, 13.60 and 13.81 eV, respectively.

The relatively low value of the adiabatic onset determined for H_2SiMe^+ (13.81 eV) and its large amount can be explained assuming that it is produced in an ion rearrangement process involving a transition from SiMe_3^+ . According to,

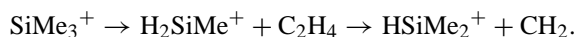


According to van Der Kelen et al. [5], the relative abundance (as percentage of the total ionisation of SiMe_4) of SiH^+ , SiH_2^+ , SiH_3^+ , HSiMe^+ , H_2SiMe^+ , HSiMe_2^+ ions, can only be explained as an ion rearrangement and their total contribution to the ionisation is very large (more than 20%). Authors [5,10,11] propose general fragmentation schemes, which involve metastable supported decomposition pathways, with H rearrangement and elimination of CH_2 or C_2H_4 from

one or two methyl groups, respectively. These schemes can be summarized as follows,



and



According to the literature, the threshold ionisation value measured for H_2SiMe^+ ranges between 13.6 and 13.8 eV [10] and 14 eV [12]. In our case the threshold ionisation measured ranges between 10 and 11.5 eV. These values are lower than the previous ones. Moreover, it remains the same when the discharge is off or on. This behaviour can be explained assuming that the H_2SiMe^+ ion produced in our case even at an electron energy lower than 13.81 eV is probably due to the ionisation of other species than SiMe_4 . This assumption is confirmed by the results given in Fig. 4.

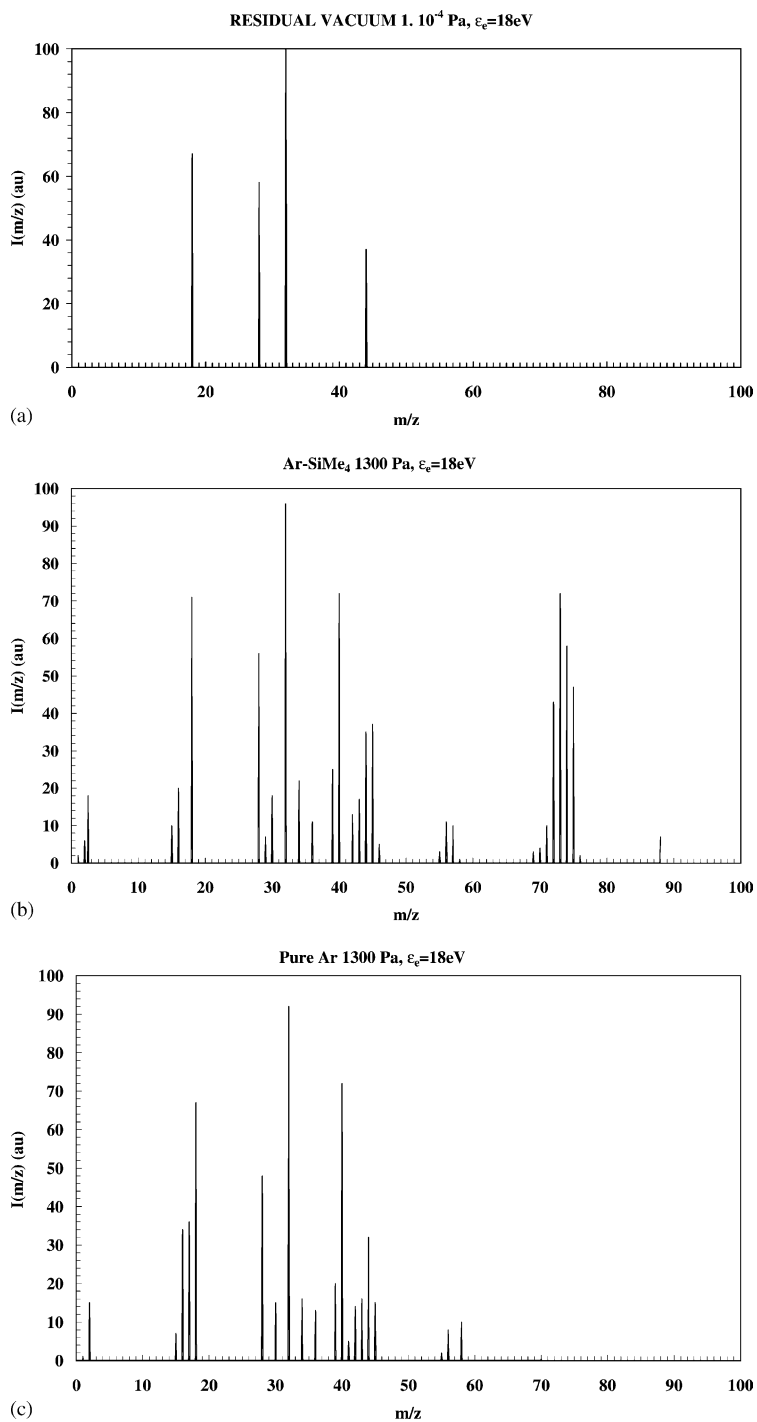


Fig. 4. Mass spectrometer spectrum measured vs. the m/z ratio at an electron energy equal to 18 eV. (a) This spectrum is performed in the residual vacuum at 10^{-4} Pa. (b) This spectrum is performed in an argon–tetramethylsilane gas mixture at 1300 Pa when the discharge is on. (c) This spectrum is performed when the tetramethylsilane has been removed in pure argon when the discharge is off.

This figure exhibits scanned bargraph data based on the relative abundance of species. The mass number ratio m/z ranges between 0 and 100. Measurements are performed with an electron energy equal to 18 eV and under three different experimental conditions. The first spectrum is performed in the residual vacuum at 10^{-4} Pa, before to inject the tetramethylsilane in the vessel. The second spectrum is performed with discharge at the same electron energy but in an argon–tetramethylsilane gas mixture at 1300 Pa. The last spectrum is obtained without discharge in pure argon at 1300 Pa, just after the former spectrum acquisition i.e., the argon–tetramethylsilane mixture is removed and the vessel is cleaned with an argon flow and feed with 1300 Pa of pure argon. These are only qualitative results because the peak intensity values are not averaged on several data acquisitions. As it can be observed in the first spectrum, only peaks at $m/z = 18, 28, 32, 44$ are observed. Because of the threshold ionisation determined for these species, they correspond probably to H_2O^+ , N_2^+ , O_2^+ , and probably to HSiMe^+ or C_3H_8^+ ions (see Table 1). When the tetramethylsilane is injected within the vessel, more peaks are observed. They are corresponding to the peaks previously related in Table 1. The main peaks are corresponding to H^+ , H_2^+ , CH_3^+ , SiMe_2^+ , SiMe^+ , SiMe_3^+ , HSiMe^+ , H_2SiMe^+ , SiMe_4^+ , C_2H_4^+ , ... When the vessel is cleaned with and argon flow and when argon is injected within the reactor without tetramethylsilane (third spectrum), the bargraph spectrum shows that the group of peaks previously observed between $m/z = 70$ and 80 is disappeared and consequently the mass $m/z = 73$ is not observed whereas the mass $m/z = 45$ still observed. These results shows that H_2SiMe^+ can be observed without SiMe_3^+ and consequently that others mechanisms that ion SiMe_3^+ rearrangement are providing H_2SiMe^+ ion. These mechanisms probably involves desorbed species from the tube wall, because H_2SiMe^+ ion is observed without tetramethylsilane injected.

Concerning the mass observed at $m/z = 58$ (SiMe_2^+ species), the threshold ionisation energy ranges between 10 and 11 eV. It should be expected

at a larger electron energy if these ions proceed only from SiMe_4 dissociation ionisation (13.9 eV). The formation of these species at low electron energy, without discharge is probably due to desorbed species from the reactor wall, as HSiMe_2 , HSiMe_3 or H_2SiMe_2 (see Table 1). This assumption agree with the fact that the peak $m/z = 58$ is also observed without tetramethylsilane injected. As it is shown on the third spectrum.

Fig. 5 shows the change in the signal measured for H_2SiMe^+ ($m/z = 45$) vs. the time. Measurements are performed in the vacuum at 10^{-2} Pa without SiMe_4 injected. The signal is slowly decreasing vs. time, 48 min after the beginning of the data acquisition it remains equal to 18.7% of the signal measured 3 min after the beginning.

Moreover, the change of the signal intensity measured for HSiMe^+ , H_2SiMe^+ or SiMe_2^+ vs. time remains lower than 5% when measurements are performed 5 min after to stop the injection of the tetramethylsilane.

Table 2 shows the relative signal intensity of SiMe_4^+ , SiMe_3^+ , SiMe_2^+ , HSiMe^+ , H_2SiMe^+ vs. the argon pressure when tetramethylsilane has been stopped 1 h before. Fig. 5 shows that in this condition, the signal intensity is expected to remain quite unchanged if the pressure is kept constant. The signal measured for SiMe_3^+ and SiMe_4^+ is very low. It is larger for HSiMe^+ , H_2SiMe^+ or SiMe_2^+ which probably are produced from desorbed species. For all the species the signal intensity decreases with increasing argon pressure. The decrease of the signal intensity value is ranges between 22 and 37% when the argon

Table 2

The change in the signal intensity of SiMe_4^+ , SiMe_3^+ , SiMe_2^+ , HSiMe^+ , H_2SiMe^+ vs. the argon flow when tetramethylsilane has been stopped

P_{Ar} (Pa)	SiMe_4^+	SiMe_3^+	SiMe_2^+	HSiMe^+	H_2SiMe^+
0.004	0.13	0.11	0.71	1.00	0.41
1	0.11	0.10	0.64	0.89	0.35
7.0	0.11	0.09	0.60	0.82	0.32
25.0	0.10	0.08	0.56	0.76	0.29
35.0	0.10	0.08	0.55	0.72	0.26

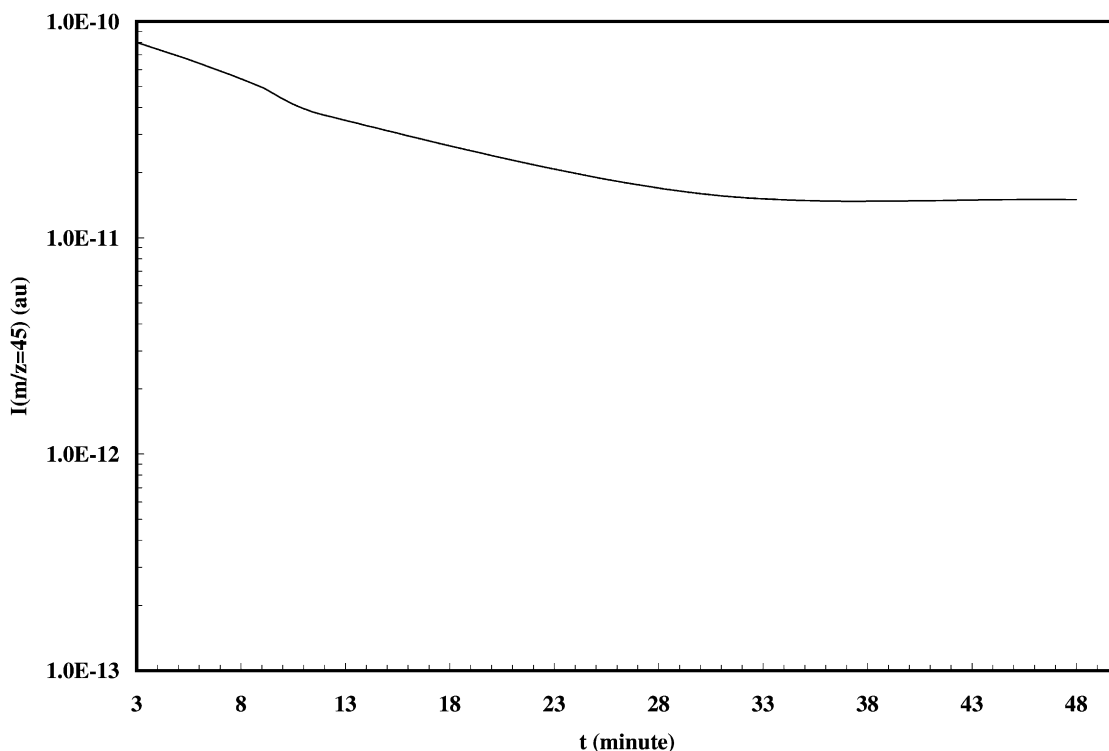


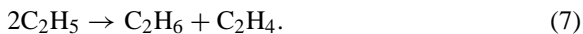
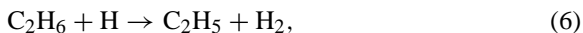
Fig. 5. The change in the signal intensity measured for H_2SiMe^+ ($m/z = 45$) vs. the time.

partial pressure is increasing from 0.004 to 35 Pa. It can be ascribed to the dilution effect of desorbed species from the wall in the argon injected within the reactor. Thus, the signal intensity measured for HSiMe^+ , H_2SiMe^+ or SiMe_2^+ remains large enough even when the tetramethylsilane is not injected. It is typically 30% of the signal measured when the tetramethylsilane is injected. This effect is amplified by the fact that in the reactor the length of the intermediate part between the mass spectrometer sample hole and the discharge exit is large enough (20 cm) to produce an important amount of desorbed species.

According to the literature [5,10,11] C_2H_4^+ is produced after ionisation of C_2H_4 which is created when the rearrangement process of SiMe_3^+ occurs. Nevertheless, others mechanisms can also provide these species. Wrobel and coworkers [13,14] have studied the pyrolysis of polymer films obtained by means of

chemical vapour deposition of tetramethylsilane. They have observed that a large amount of methane, ethane or ethylene is evaporated because of the thermal decomposition of silylmethyl group. These authors explain this behaviour assuming that these species are produced in the recombination processes of methyl groups and atomic hydrogen in the vessel. The density of methyl radicals or H atoms accounts for dissociation processes of Si–C and C–H bond breaking. Me groups and H atoms recombine in efficient processes producing methane ethane or ethylene, as it follows [13,14],



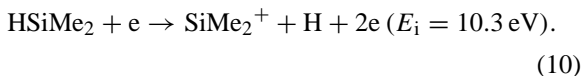
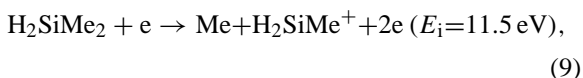
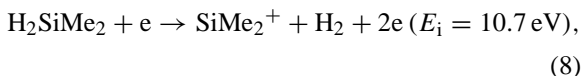


In these mechanisms, C_2H_5 is assumed to be unstable.

Assuming these former reaction processes, C_2H_4^+ can proceed via the dissociation ionisation of C_2H_6 , the threshold ionisation is equal to 12.08 eV (see Table 1). C_2H_6 is formed in the reaction (3). It can also proceed via the ionisation of C_2H_4 produced in reactions (4) and (5). The threshold ionisation of C_2H_4 is 10.5 eV (see Table 1). Consequently, C_2H_4^+ ions can also be observed in the reactor when SiMe_3^+ ion rearrangement process are not produced. Similar mechanisms can occur in an electrical discharge because of a deposited film produced on the reactor wall and due to the decomposition of the tetramethylsilane. These thin films can produce a large amount of various desorbed species as Me groups or H atoms.

H_2SiMe^+ , HSiMe^+ , SiMe_2^+ and C_2H_4^+ ions probably proceed via ionisation of desorbed species from the reactor wall.

These species could be H_2SiMe_2 or HSiMe_2 , they can be dissociated by electron impact in the mass spectrometer ionisation chamber as it follows [9],



If we compare the signal intensity values measured for both species SiMe_2^+ and H_2SiMe^+ , assuming that the transmission factor of the mass spectrometer analyser and that the ionisation cross-section of both species are the same [6], the mass spectrometer signal intensity ratio for the two species is equal to the density ratio of the species. Typically, $I(m/z = 45)/I(m/z = 58) = 1.27$. Consequently, the desorbed species are mainly producing H_2SiMe^+ (reaction (9)).

As previously explained C_2H_4^+ ions is expected to be produced because of the recombination processes of methyl groups and H atoms desorbed by the reactor wall and in this case it is not produced in rearrangement process of SiMe_3^+ ion.

4. Conclusion

This work is devoted to the study of H_2SiMe^+ , HSiMe^+ and SiMe_2^+ ions produced in a microwave discharge containing an Ar– SiMe_4 gas mixture.

We have shown by means of a mass spectrometer and using of the threshold ionisation method that H_2SiMe^+ , HSiMe^+ ions are not produced via the SiMe_3^+ ion rearrangement process proposed in the literature [5,10,11], but result probably of the ionisation of desorbed species from the reactor wall. These species could be H_2SiMe_2 or HSiMe_2 . The low threshold ionisation value determined for SiMe_2^+ shows that this ion is also probably produced by the ionisation of desorbed species and not only from the SiMe_4 dissociation ionisation. From the literature study, we show that C_2H_4^+ which is supposed to be produced in the SiMe_3^+ ion rearrangement process can also be produced by methyl groups and H atoms recombination processes.

Consequently, these results show the important role of desorbed species from the reactor wall, which can act as a complementary source of species injected within the plasma.

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