

**KINETICS OF HYDROCARBON FORMATION  
IN a-C:H FILM DEPOSITION PLASMAS**

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## INTRODUCTION

A considerable effort has been recently devoted to the modelling of the growth of hydrogenated carbon films (a-C:H), widely used for first wall conditioning of fusion devices nowadays [1], from the gas phase in PACVD of methane and its admixtures with  $H_2$  [2-4]. Quite surprisingly, although the transport of radicals and ions to the growing film has been considered in the simulation of the observed carbon deposition rate [4], the chemical interaction between the gas phase and the film to yield some of the detected species included in such simulations has been neglected. On the other hand, the formation of hydrocarbons and radicals during the erosion of several types of carbon substrates, including a-C:H films, by H atoms and H and noble gas ions has been extensively reported either in beam-solid interactions [5-7], glow discharges [8] and fusion plasmas [9].

In the present work, the mass spectrum of neutral species has been used to characterize the formation mechanism of  $C_2$  and  $C_3$  hydrocarbons in PACVD of admixtures of methane with  $H_2$  and He under several deposition conditions and a simple model for the formation kinetics of the observed species is proposed.

## EXPERIMENTAL

The experimental set-up has been described in previous papers [8,10]. Briefly, a DC glow discharge of admixtures of methane with hydrogen and helium at a total pressure of  $10^{-2}$  torr is produced in a SS chamber, pumped by a 450 l/s turbo pump (base pressure  $< 2 \times 10^{-7}$  torr) at room temperature. In the experiments here reported, the methane content in the admixtures was varied from 1 to 15 %. The chamber is grounded and acts as the cathode of the discharge, the total area exposed to the plasma being  $\approx 6000 \text{ cm}^2$ . The pumping speed for several gases ( $H_2$ , He,  $CH_4$ ,  $C_2H_2$ ,  $N_2$  and  $CO_2$ ) were measured at the pressure of interest by injecting a fast pulse of gas and recording the time decay by a fast capacitance manometer.

The gas species during the discharge are analyzed using a differentially pumped RGA (Balzers QMG-125). The base pressure in the quadrupole chamber is  $< 10^{-9}$  torr and the rhenium filament was conditioned before the experiments by heating it in a hydrogen background. The absolute calibration for the gases above mentioned was

performed at several ranges of chamber pressure (  $10^{-5}$  to  $2 \times 10^{-2}$  torr ). The calibration factors for other reported species were obtained by considering the observed mass effect on the transmission through the tube connecting both chambers and the tabulated total ionization probabilities. The observed spectra were corrected for the effect of hydrocarbon formation in the absence of plasma and fed into a computer program. The procedure followed by J. W. Davis *et al* [7] was used to reconstruct the gas composition from the mass spectra.

Deposition rates were inferred from the total balance of C atoms in the discharge, mainly given by the depletion of methane after the plasma is switched on [10]. The interference colours method [11] was eventually used as a reference of the film thickness. Plasma optical emission in the visible range was also used, specially to characterize the metallic wall through the emission of Cr atoms and CO molecules under He bombardment. The mass spectra during the etching in a pure  $H_2$  plasma of the deposited films between experiments ( see below ) were also used as a reference of initial wall conditions.

## RESULTS

The time evolution of the mass spectra recorded after the initiation of the glow discharge in the admixtures above mentioned indicate the existence of several distinct phases, depending on the amount of C atoms deposited on the wall. In what follows, we shall describe the experimental findings according to these phases.

During the initial phase of the experiment ( phase a ), a continuous decrease of peaks at  $M/e=28$  and 44 and an increase of those at  $M/e=26$ , 27 takes place, the remaining peaks staying basically constant during this period. The total amounts of C atoms deposited during this phase are  $2 \times 10^{16}$  and  $1.6 \times 10^{16} \text{ cm}^{-2}$  for the  $H_2$  and He plasmas, respectively. Figure 1 shows the evolution of the partial pressures of  $C_2$  hydrocarbons and C oxides during this phase for the case of He/ $CH_4$  ( fig. 1a ) and  $H_2/CH_4$  ( fig. 1b ) admixtures for the same conditions of plasma current, total pressure and gas composition. In both cases a strong, fast increase of ethylene and a, slower, decrease of CO and  $CO_2$  are clearly observed. Also, a slower rise and a higher asymptotic level of ethylene are observed in the case of the hydrogen admixture. The change of the plasma conditions (  $V \approx 400 \text{ V}$  for both plasmas ) and of the partial pressure of methane in the plasma was

negligible during this phase of the experiment. At longer times ( phase b ) , corresponding to C fluences up to  $\approx 10^{17} \text{ cm}^{-2}$ , all the peaks reach a constant value , except those at  $M/e=28$  ( CO ) and 44 ( CO<sub>2</sub>), that keep decreasing. This behaviour was clearly observed in H<sub>2</sub>/CH<sub>4</sub> plasmas but some increase of the acetylene partial pressure was seen in those produced from He admixtures during this phase.

For C fluences above  $\approx 10^{17} \text{ cm}^{-2}$  ( phase c ) a strong increase of the partial pressure of acetylene in gas mixture takes place, eventually reaching a constant value at longer times ( 4800 and 2300 seconds for the H<sub>2</sub> and He plasmas, respectively ) , while the partial pressures of other hydrocarbons stay constant ( figure 2 ). A small decrease in the plasma voltage (  $\approx 350\text{V}$ ) was observed during the time corresponding to the rise of acetylene, suggesting a change in the plasma conditions as the film grows up. As seen in the figure, a faster rise of acetylene is observed in the He plasma. A summary of the asymptotic values for the partial pressures of C<sub>2</sub> and C<sub>3</sub> hydrocarbons obtained under the different plasma conditions here studied is given in Table 1.

It should be mentioned that, although the different phases described above were observed under most of the deposition conditions reported, for very low methane concentration in the gas mixture ( $< 2\%$  ), the last phase was not reached even after C fluences twice as large as the expected critical one. When the mass spectrum was recorded during the etching of the film in a pure H<sub>2</sub> plasma a mirror-like situation was obtained, namely the acetylene started to decrease before other hydrocarbons ( methane and ethylene ) did as the film was being etched away.

As shown in table 1, the cracking of methane (  $\Delta P_{\text{CH}_4}$  ) strongly depends on plasma conditions . For the dominant species, acetylene and ethylene, a fair proportionality between the concentration of these species in the plasma and the deposition rate, directly proportional to the depletion of C containing molecules in the gas phase, is seen for constant plasma current. This result was confirmed by slowly increasing the concentration of methane in these plasmas at constant current ( 250 mA ). In order to compare the relative hydrocarbon production yield in the different gas mixtures ( H<sub>2</sub> vs. He ) the partial pressures of the C<sub>2</sub> and C<sub>3</sub> hydrocarbons are plotted in figure 3 for the same value of  $\Delta P_{\text{CH}_4}$ . As shown, a much higher yields of ethylene and ethane and a lower one for C<sub>3</sub>H<sub>6</sub> are obtained in the H<sub>2</sub>/CH<sub>4</sub> plasma. In order to check the influence of the different film growing conditions on the hydrocarbon production, a

He/CH<sub>4</sub> plasma was initiated on a film grown from a H<sub>2</sub>/CH<sub>4</sub> admixture. The relative hydrocarbon yields obtained after one minute of plasma exposure were very similar to those characteristics of the experiments in He/CH<sub>4</sub> plasmas.

## DISCUSSION

There is a surprising similarity between the phases of hydrocarbon production here reported and the behaviour of the C Auger peak during the film growth by direct ion beam deposition on W at room temperature reported by Tscherich [12]. For a C<sup>+</sup> flux of 10<sup>14</sup> cm<sup>-2</sup> s<sup>-1</sup>, he found a rise of the surface (0.7 nm) C concentration followed by a phase where this concentration remained constant, while diffusion into the metal increased the C content by metal carbide formation. The total fluences at which these phases took place were 3x10<sup>16</sup> and 10<sup>17</sup> cm<sup>-2</sup>, respectively, that correspond in our case to the initial rise of ethylene and its stabilization before the rise of acetylene formation. The duration of phase c in our case is also very similar to the time required for the growing of the first nanometer of a-C:H film on top of the metallic carbide (≈ 3000 s) in Tscherich's work. Since the projected range of implantation of 300 eV C ions in the film is ≈ 2nm, this result strongly suggest the the formation of acetylene molecules by the impact of C ions( CH<sub>x</sub><sup>+</sup> in general ) with the film, this mechanism being at least 10 times more efficient than gas phase radical reactions, as indicated by the detected level of acetylene before phase c. Assuming that the minimum possible flux of C ions correspond to the carbon deposition rate ( 7x10<sup>13</sup> cm<sup>-2</sup> s<sup>-1</sup> for the conditions of fig. 1b ), a maximum yield of 0.06 molecules/ion( 0.08 if cracking of C<sub>2</sub>H<sub>2</sub> in the plasma is taken into account ) , that for a 2 nm interaction range ( ≈ 1.6x10<sup>16</sup> C/cm<sup>2</sup>) correspond to a maximum cross section of ≈ 0.5x10<sup>-17</sup> cm<sup>-2</sup>. This yield is about a factor of 2-3 larger than that corresponding to the sputtering of the film by He ions to form the same hydrocarbon and ≈ 6 times larger than for H ions in similar conditions [8].

The transient behaviour of the ethylene partial pressure during phase a has no unambiguous interpretation. The main source of this hydrocarbon in methane plasmas has been ascribed to the gas phase reaction between CH radicals and CH<sub>4</sub> molecules [2-4]. One might speculate that the carburization of the plasma facing surface could strongly affect the sticking probability, S , of CH radicals, the lower probability in the carbide



corresponding to a higher concentration in the gas phase. A simple kinetic model, taking into account the formation of CH radicals by electron impact and its losses through chemical reactions (mostly with methane) and diffusion, similar to that used in the bibliography [2-4], indicates however that under the conditions here reported a change in  $S$  from 1 to 0.1 (metal-carbide) will have a much smaller effect in the formation rate of ethylene than observed. Also, a stronger dependence on the concentration of methane and a lower yield would be expected.

At this point, it is interesting to note the lower yield for ethylene and the higher one for  $C_3H_6$  in the He plasmas compared to those in  $H_2$ . This difference could be simply explained by the competitive reaction between H and  $CH_3$  radicals with  $\cdot CH=CH_2$ . Since these reactions are highly exothermic, the recombination of these radicals will be favoured on the surface.

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**Table 1**

Summary of hydrocarbon production in a-C:H deposition plasmas

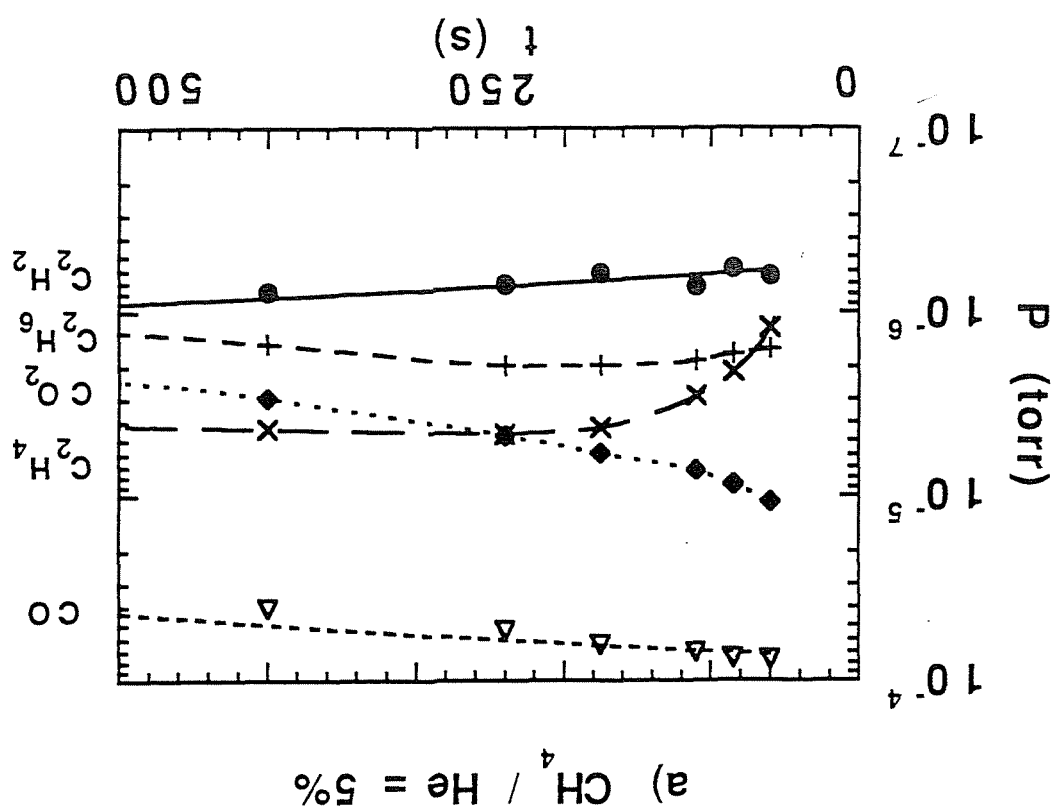
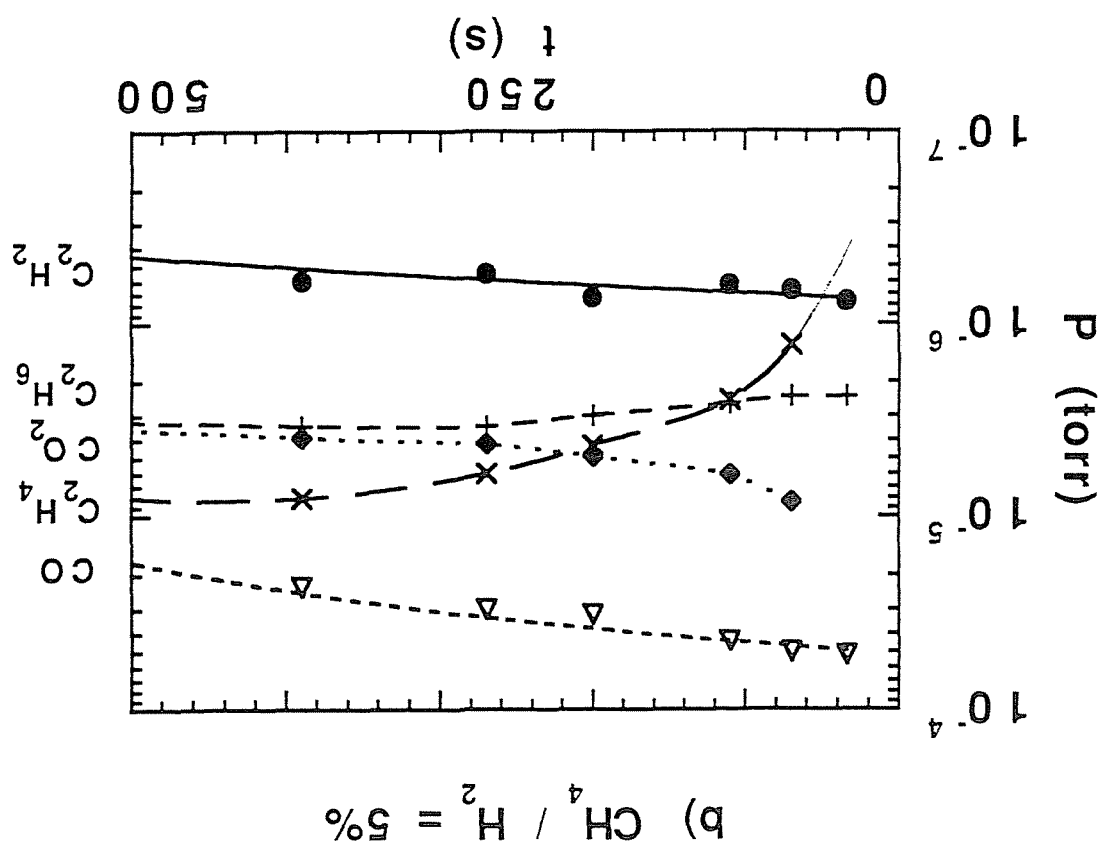
Mixture		partial pressures ( $10^{-6}$ torr)					
(Gas, %CH <sub>4</sub> )	I <sub>p</sub> (mA)	$\Delta P_{CH_4}$ ( $10^{-4}$ torr)	C <sub>2</sub> H <sub>2</sub>	C <sub>2</sub> H <sub>4</sub>	C <sub>2</sub> H <sub>6</sub>	C <sub>3</sub> H <sub>6</sub>	C <sub>3</sub> H <sub>8</sub>
H <sub>2</sub> , 5	250	1.4	7.0	5.4	0.7	-	0.4
H <sub>2</sub> , 10	250	2.0	11.0	8.0	1.8	0.55	0.6
H <sub>2</sub> , 15	500	6.4	23.0	9.4	1.1	2.0	0.5
He, 5	250	1.9	8.0	2.6	0.41	1.6	0.2
He, 10	250	3.5	17.0	7.1	1.5	1.0	0.7
He, 3.5	500	1.7	3.8	2.1	0.54	0.42	0.32

## FIGURE CAPTIONS

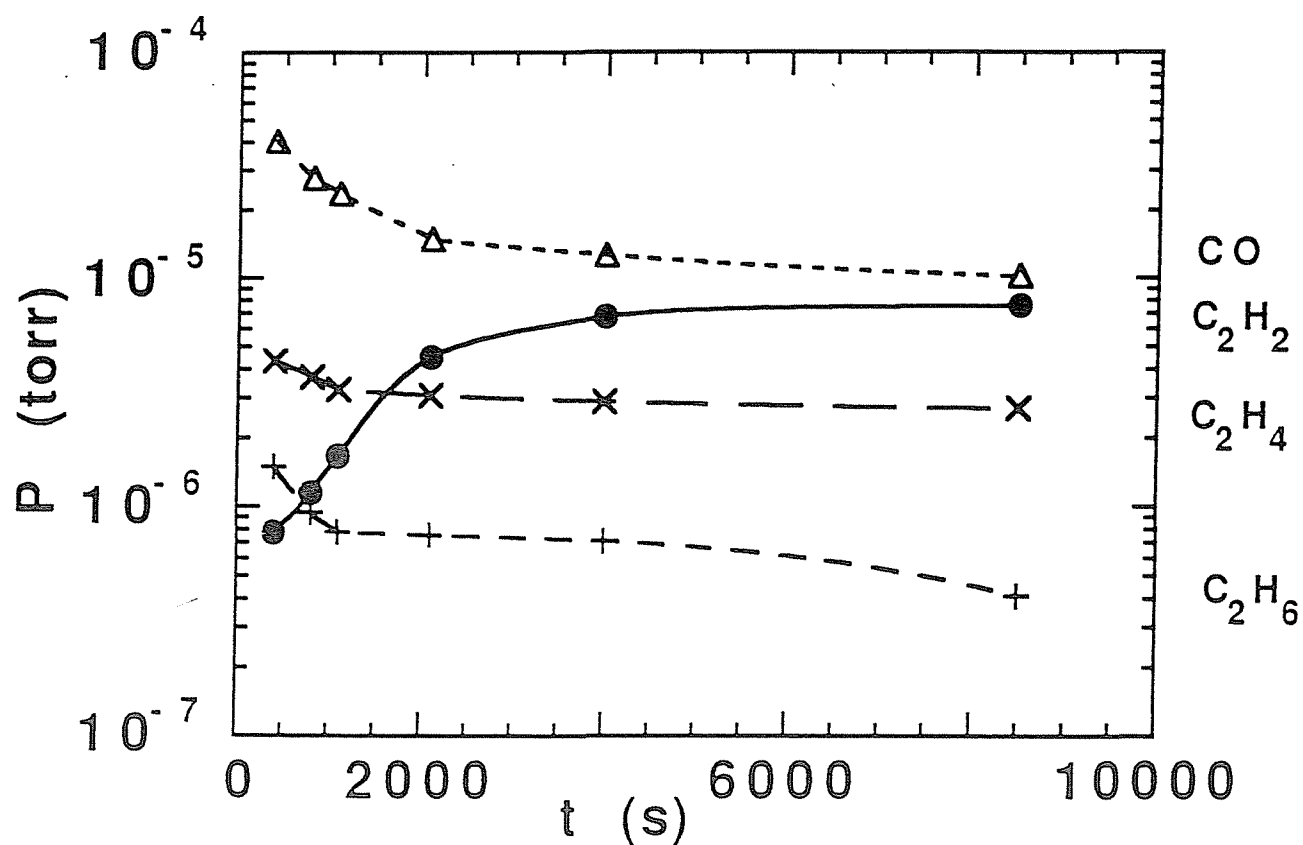
**Figure 1.** Time evolution of the carbon oxides and  $C_2$  hydrocarbon partial pressures during the initial phase of the experiment at 250 mA and a total pressure of  $1 \times 10^{-2}$  torr for two different methane/buffer admixtures.

**Figure 2.** Same as in figure 1 for longer times.

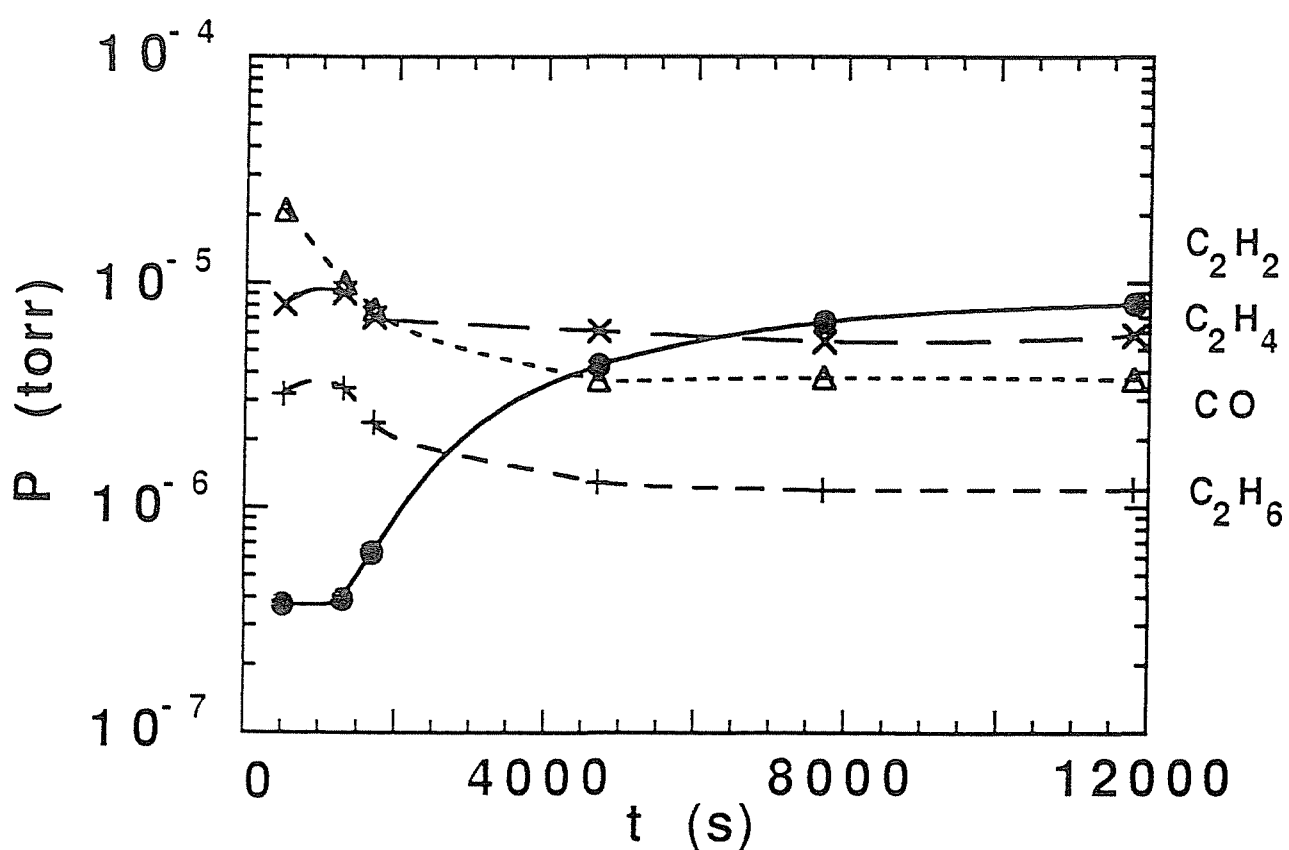
**Figure 3.** Comparison between the detected asymptotic partial pressures of  $C_2$  and  $C_3$  hydrocarbons in  $H_2/CH_4$  and  $He/CH_4$  plasmas at constant deposition rate.

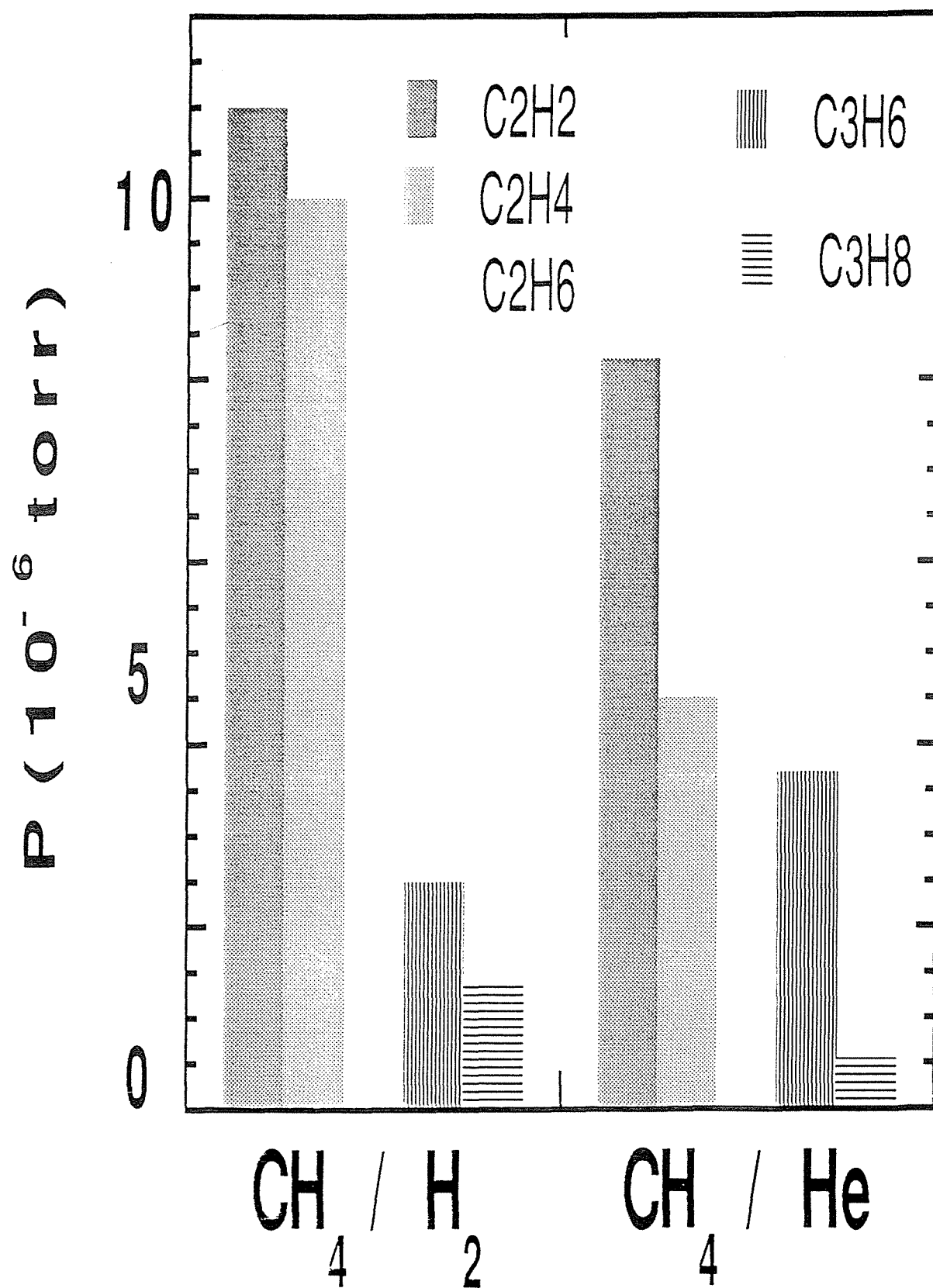


a)  $\text{CH}_4 / \text{He} = 5\%$



b)  $\text{CH}_4 / \text{H}_2 = 5\%$









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20 pp., 3 fig., 12 ref.

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Se han investigado los mecanismos de formación de los hidrocarburos tipo C2 y C3 durante la deposición asistida por plasma (PACVD) a partir de mezclas de metano con hidrógeno y helio. Los estudios se han realizado por espectrometría de masas para diversas condiciones de la deposición. La evolución temporal de las especies observadas indica que la formación de etileno y acetileno depende sensiblemente de las condiciones de la pared (metálica, carburo, film) durante la deposición. De acuerdo con los resultados obtenidos, la formación de acetileno está directamente relacionada con el crecimiento de la película de carbono sobre el carburo metálico previamente formado.

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