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and G.A. Somorjai

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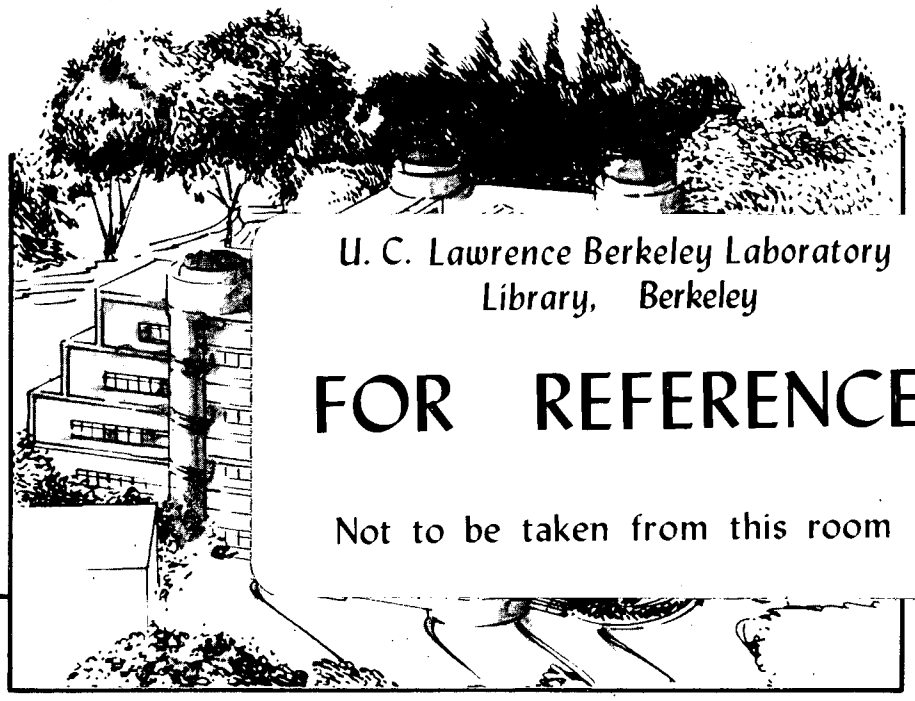

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Abstract

Hydrogenated amorphous silicon carbide ($Si_xC_yH_z$) films were synthesized by plasma enhanced chemical vapor deposition using monomethylsilane (CH_3SiH_3) as the precursor. Silicon (100) wafers and gold foils were employed as substrates. A mass spectrometric analysis of the plasma showed that the advantage of using monomethylsilane relative to a silane/hydrocarbon mixture is that the majority of the Si-C bonds were preserved in the CH_3SiH_3 plasma.

The composition and the morphology of the $Si_xC_yH_z$ films was studied via X-ray Photoelectron Spectroscopy, Auger Electron Spectroscopy, and Scanning Electron Microscopy as a function of the substrate temperature, composition of the ion flux bombarding the surface and kinetic energy of these ions. The oxygen content of the films was found to decrease monotonically with increasing substrate temperature.

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1. Introduction

The technological potential of silicon carbide has resulted in the development of several fabrication methods of SiC (1-3). Crystalline films can be produced by chemical vapor deposition (CVD) (4-10), but the relatively high substrate temperature (T>1270K) required in this process limits its field of application. Radio frequency sputtering (11) and plasma enhanced chemical vapor deposition techniques, which do not require such high temperature, produce mainly amorphous samples. Deposition of SiC by glow discharge (plasma) has generally used SiH_4/hydrocarbon (12-24), SiH_4/hydrocarbon/noble (25,26) and SiH_4/hydrocarbon/H_2 (27-29) gas mixtures as feedstocks. To our knowledge, few other silicon compounds have been used in these types of experiments (30,31). Tetramethylsilane (32-34), monomethylsilane (35) and disilylmethane (36,37) are the most common, but they were mixed with silane and were regarded as the carbon source rather than the source of silicon and carbon. Patents of the Shinetsu Chem. Co. Ltd. (38) and Sanyo Elect. (39) describe the preparation of SiC by various methods from complex silicon compounds.

In contrast with previous approaches, this paper deals with the synthesis of silicon carbide films by PECVD from a pure monomethylsilane feedstock gas. The films were deposited onto polycrystalline gold foils and silicon (100) wafers chosen for adhesion considerations. The composition of the plasma was examined by mass spectrometry. The structure and the composition of the SiC films produced was studied as a function of substrate temperature (333-723 K) and ion bombardment. The properties of these films are compared with SiC produced under different conditions and by other techniques.

2. Experimental procedure

The r.f. plasma system has been described in detail elsewhere (40). Therefore, here we only mention the main features of the equipment that are relevant for the presented results. The r.f. electrodes consisted of two horizontal stainless steel disks separated by a gap of two centimeters. Either electrode could be powered by a 13.56 MHz r.f. generator, the other electrode being grounded. The films were deposited on the lower electrode which could be resistively heated as high as 773K, even when powered.The development of a self-bias, due to the difference in the mobility of the electrons and the ions, was observed at the powered electrode. For a r.f. power of 50W and a CH_3SiH_3 working pressure of 13.3Pa, a self-bias of 275V was measured. The monomethylsilane was introduced via an orifice in the center of the upper electrode. The stainless steel reactor was pumped down by a rotary vacuum pump and a sorption pump (base pressure $=1.3 \times 10^{-2}$ Pa).

The plasma chamber was connected through a differentially pumped middle stage to a high vacuum chamber in which was installed a UTI model 100C mass analyser along with ion deflection lenses to measure the ion energy distribution. The first orifice through the lower electrode in the plasma chamber was approximately 100µm in diameter, while the second orifice between the middle stage and the lower high vacuum chamber, was about 1mm in diameter. Pressure was maintained on the order of 10^{-6} Pa in the lower chamber. In the ionizer-off operation mode, every component in the ionization chamber was grounded except for an ion focusing lens and only ions were detected. The mass spectra of the neutral species were taken under the normal ionizer-on operation mode with electron energy of 75 eV and ion energy of 19 eV.

Two types of substrates were used in the present study : boron doped Si (100) wafers and gold foils. Silicon was choosen in view of the good results obtained by CVD on this substrate. In both case there is a possible formation of chemical bonds between the substrate and the deposit : gold could form a silicide and silicon could form Si-Si and Si-C bonds at the interface. Before being introduced into the chamber for deposition, the Si (100) wafers were etched for five minutes in 49% HF in order to remove the native oxide layer . The gold foils were prepared for deposition by polishing with 0.05mm alumina paste and rinsing with acetone and methanol. Once in the deposition chamber, the surfaces of the substrates were further cleaned by argon

bombardment, followed by ignition of the CH_3SiH_3 plasma.

The crystal structure of the deposited silicon carbide films was examined by X-Ray Diffraction (XRD) using a Siemens diffractometer. The surface composition and morphology of the films was determined using X-ray Photoelectron Spectroscopy (XPS), Scanning Auger Electron Spectroscopy (SAES) and Scanning Electron Microscopy (SEM). The instruments used were a PHI 5300 ESCA system and a PHI 660 Scanning Auger Multiprobe. Argon depth profiling was performed in the XPS and AES apparatus with an eroded area of $1cm^2$ and $1mm^2$, respectively. The ions energies were 3 and 5 keV respectively. The concentrations indicated in the following sections were thus representative of the bulk composition. The thickness of the films was determined by either profilometry (films thicker than 200nm) or ellipsometry (films thinner than 200nm).
The main impurities of the gas itselfs (purity 97%) were, following the producer, methyldichlorosilane and methylchlorosilane.

3. Results and discussion

I. Mass spectrometric study of the species in a CH_3SiH_3 plasma.

A. Neutral species

The neutral species impinging on both the powered and the grounded electrodes were measured at two different pressures of CH_3SiH_3 (7 and 13.3 Pa), and at three different r.f. powers (50, 100 and 200W). As figure1 shows, the main peak in the mass spectrum appeared at 44 amu, which corresponds to a monomethylsilane molecule deficient of two hydrogen atoms. Three structures could be assigned to the 44 amu fragment : CH_3SiH, CH_2SiH_2, and $CHSiH_3$. Experiments with a deuterated monomethylsilane (CD_3SiH_3 or CH_3SiH_3) would allow one to unambigously assign a structure to this 44 amu neutral fragment. Also, a considerable amount of H_2 and traces of CH_4 and SiH_4 were detected. A comparison of figures 1 and 2 shows that plasma ignition produced minimal changes in the CH_3SiH_3 mass spectra. The most obvious change induced by plasma ignition was an increase in the amount of hydrogen (H_2, H). We concluded that the plasma did not break the majority of the C-Si bonds as the ratio $CSiH_z$/SiHy did not vary significantly in the absence or presence of.the plasma (3.4 and 3.5 respectively). In the plasma, the radicals responsible for the deposition process were most probably the $CSiH_z$ ($0 \leq z \leq 5$) radicals produced by the breaking of C-H and Si-H bonds. No noticeable variation in plasma composition with

the CH_3SiH_3 pressure was observed. No molecular species with mass higher than 46 amu was detected. The proportions of fragments and intact molecules were the same at both electrodes (powered and grounded).

The predominance of gas phase species containing a Si-C bond in the CH_3SiH_3 plasma is a major advantage for the formation of silicon carbide films via a PECVD process. For comparison, a mass spectrometric analysis of a r.f. discharge of a 1:1 CH_4/SiH_4 mixture produced in the same apparatus, showed that few Si-C bonds were created in the CH_4/SiH_4 plasma itself. The Si-C bond must be formed, in this case, at the surface of the substrate, in agreement with the observations of Catherine et al. (41). Therefore, the probability of obtaining Si-C bonds in the deposited films should be higher in the case of a monomethylsilane plasma versus a CH_4/SiH_4 plasma. In the CH_3SiH_3 plasma, the absence of molecular species with mass greater than 46 amu indicates a very low rate of polymerization in the gas phase.

B. Positive ion species

The ions species were investigated under the same conditions of pressure and r.f. power as were the neutral species. The ions detected in the CH_3SiH_3 plasma included the following :

$$CH_x^+ \ (2 < x < 4),\ SiH_y^+ \ (2 < y < 4)\ ,\ \text{and}\ SiCH_z^+ \ (3 < z < 5).$$

The ions contributed 0.1% to the total flux of particles impinging the electrode surface.

As expected, the mass spectrum of ions depended strongly on the electrode examined (fig.3). The average degree of hydrogenation of Si and Si-C species was lower at the powered electrode than at the grounded one. More ions containing the Si-C bonds bombarded the grounded electrode than the powered one. The difference in the ion flux composition between the grounded and the powered electrode can be attributed to the influence of the self-bias voltage that developes spontaneously at the powered electrode. Due to the potential drop in the vicinity of the powered electrode, the average electron-molecule collision energy is higher and the impact is more likely to break the Si-C, Si-H and C-H bonds, thus increasing the fraction of light and less hydrogenated ions.

II. Film composition and structure

A. influence of the substrate temperature during film deposition

The four substrate temperatures investigated were 333, 423, 573 and 723 K.

The resulting dependance of the deposition rate (thickness per unit deposition time) on the substrate temperature is given in table 1. The deposition rate decreased by a factor of four for an increase in temperature of 400 K. A similar behaviour was observed with films deposited on gold.

i. Grounded electrode

The XPS and AES analysis showed that the composition of the films deposited on gold at the grounded electrode depended dramatically on the substrate temperature during deposition. At 333 and 423 K, the films deposited at the grounded electrode were heavily contaminated with oxygen while films deposited at 573 and 723 K contained less than 1 % of this element (see table 2). This oxygen contamination prevented the AES analysis of these low temperature samples because of charging effects. The Si 2p peaks in the XPS spectra of these same samples were broad and contained many contributions resembling non-stoichiometric and stoichiometric oxides of silicon.A difference in the temperature dependence of the sticking coefficient of oxygenated species and Si-C species could explain the lower contamination level of the films deposited at high temperatures. The oxygen was integrated into the film during deposition and was not due to a contamination layer present at the substrate surface prior to the deposition. The source of this oxygen was probably water vapor present in the reactor and coming from the degazing of the different parts of the chamber under heating. A peak at mass 18 amu was detected in nearly all the mass spectra of CH_3SiH_3.

ii. Powered electrode

The relevant results are presented in table 2. At the powered electrode, the oxygen contamination was very low at all temperature. The main difference between the two electrodes is that a several hundred volt negative self-bias is developed at the powered electrode but not at the grounded one. This self bias voltage may remove oxygen via two routes. It is well known that in dc sputtering, a small bias voltage applied to the substrate limits the contamination of the deposit by inducing the desorption of weakly bonded species. Secondly, the growing film at this electrode is subjected to the bombardment of energetic particles. The presence of energetic hydrogen atoms (resulting from the neutralization of energetic H^+ ions) in the flux bombarding the powered electrode can induce the reduction of the oxygenated species.

Analysis of films, grown on both the powered and grounded electrodes showed that the Auger fine structure and the XPS binding energies of C and Si were

characteristic of a mixture of SiC and amorphous carbon. In XPS, the C 1s binding energy clearly showed the presence of two contributions ; carbide and amorphous carbon (fig.4). On the other hand, the silicon transitions in AES and XPS showed mainly carbidic features (fig.5). During the XPS analysis , the samples were partly covered by a gold foil, allowing to correct the energies in such a way that the $4f_{7/2}$ transition of gold appeared at 83.8 eV. The values obtained for the SiC phase of the films were slightly higher than those measured for a SiC single crystal in the same apparatus (Si 2p 100.0eV and C 1s 283.0eV). Nevertheless the values for the films remained in the error range of the apparatus and of the fitting procedure. The shift of the silicon 2p transition from a pure silicon position to a SiC position (1.2eV) was in good agreement with published values (9). The results indicated the presence of two phases in the produced films : one characterized by a majority of Si-C bonds and the other by C-C and/or C-H bonds. The 1s binding energy of the latter type of carbon did not allow the determination of the exact composition of the phase. In AES, the C KLL transition exhibited "carbide" satellite peaks at 249 and 255 eV along with a satellite peak at 240eV characteristic of amorphous carbon and graphite.

Both XPS and AES indicated that the SiC phase of the films was enriched in silicon in comparison with a stoichiometric single crystal of α -SiC analyzed under the same conditions. A difference between the sticking coefficients of the carbon-containing and the silicon-containing species could be responsible for this non stoichiometry.

The difference in composition between the films deposited at the powered electrode and those deposited at the grounded electrode was small but detectable : the powered electrode films systematically contained more silicon. The energetic hydrogen atom bombardment at the powered electrode could provoke the preferential removal of C atoms from the growing film as CH_4, with the silicon fragments being more likely to remain at the surface (32).

B. Structure

The SEM image of the films surface deposited on silicon and on gold duplicated the substrate roughness and at the observed scale (down to 10 nm), did not show any features of its own. Two factors could explain the absence of additional features in the structure. First, the electron emission of the different regions is similar. Secondly, the structures were too small for the resolution of our SEM. X-ray diffraction analysis indicated an amorphous structure for all deposition temperatures.

4. Summary and conclusions

The results obtained in this study can be summarized as follows :

-Amorphous $Si_xC_yH_z$ films can be prepared by PECVD from monomethylsilane.

-The presence of two phases is detected by AES and XPS :

1) a SiC phase rich in silicon

2) an amorphous carbon phase

-The deposition rate in terms of thickness per unit time decreases strongly with increasing substrate temperature.

-The mass spectrometric analysis of the plasma clearly shows that the majority of the C-Si bonds are preserved in the discharge, increasing the probability to include this type of bond in the films.

-The self-bias voltage present at the powered electrode increases the fraction of C-Si, C-H and Si-H bond breaking and so increases the proportion of light ions (H^+, CH_3^+...) bombarding the surface.

-Oxygen contamination of the films was observed at the grounded electrode at low temperature (333 and 423 K). A difference in the temperature dependance of Si-C and of oxygen species sticking probabilities could explain the low level of oxygen concentration in the films deposited above 423K.

-The absence of oxygen impurities in the films deposited at the powered electrode is probably a consequence of the energetic ion bombardment that induces desorption and/or reduction of the oxygenated species.

Monomethylsilane appears to be a promising precursor for the formation of $Si_xC_yH_z$ films by PECVD.

Optical and mechanical properties of these films and the influence of deposition parameters (power, pressure) are presently under investigation.

Acknowledgement

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Substrate temperature (K)	Deposition rate (nm/min)
333	22
423	20
573	10
723	6

Table 1 : Variation of the deposition rate as a function of the Si substrate temperature.
r.f. power = 50W, CH_3SiH_3 pressure= 13.3Pa

	GROUNDED ELECTRODE					POWERED ELECTRODE				
T (K)	Si 2p BE (eV)	C 1s BE (eV)	Si XPS (at.%)	O XPS (at.%)	Si AES (at.%)	Si 2p BE (eV)	C 1s BE (eV)	Si XPS (at.%)	O XPS (at.%)	Si AES (at.%)
333	98.5 102.0	284.6 280.7		15		100.0	284.8 283.2	60	<1	62
423	99.9	284.7 283.0	48	10		100.0	284.6 283.1	65	<1	65
573	100.1	284.8 283.3	56	<1	60	100.2	284.6 283.2	65	<1	60
723	100.1	284.6 283.1	63	<1	59	100.1	284.7 282.9	70	<1	62
single crystal α-SiC	99.9	283.3	50	0	50	99.9	283.3	50	0	50

Table 2 : XPS and AES analysis of films deposited on gold foils.
r.f. power = 50W, CH_3SiH_3 pressure = 13.3 Pa
Si 2p BE = Si 2p binding energy obtained by curve fitting
C 1s BE = C 1s binding energy obtained by curve fitting
Si XPS, AES = silicon concentration obtained by XPS and AES
O XPS= oxygen concentration obtained by XPS

FIGURE CAPTIONS

fig.1 : Mass spectrum of the neutral species collected at the grounded electrode.
CH_3SiH_3 pressure = 15.7 Pa, r.f. power = 100 W

fig.2 : Mass spectrum of monomethylsilane in absence of plasma
CH_3SiH_3 pressure = 6.3 Pa

fig.3 : Mass spectra of the ions collected : a) at the grounded electrode
b) at the powered electrode
CH_3SiH_3 pressure = 15.7 Pa , r.f. power = 100 W

fig.4 : Film deposited at 723 K at the powered electrode on gold.
CH_3SiH_3 pressure = 13.3 Pa, P = 50 W
C 1s photoelectron line

fig.5 : Film deposited at 723 K at the powered electrode on gold.
CH_3SiH_3 pressure = 13.3 Pa, P = 50 W
Si 2p photoelectron line

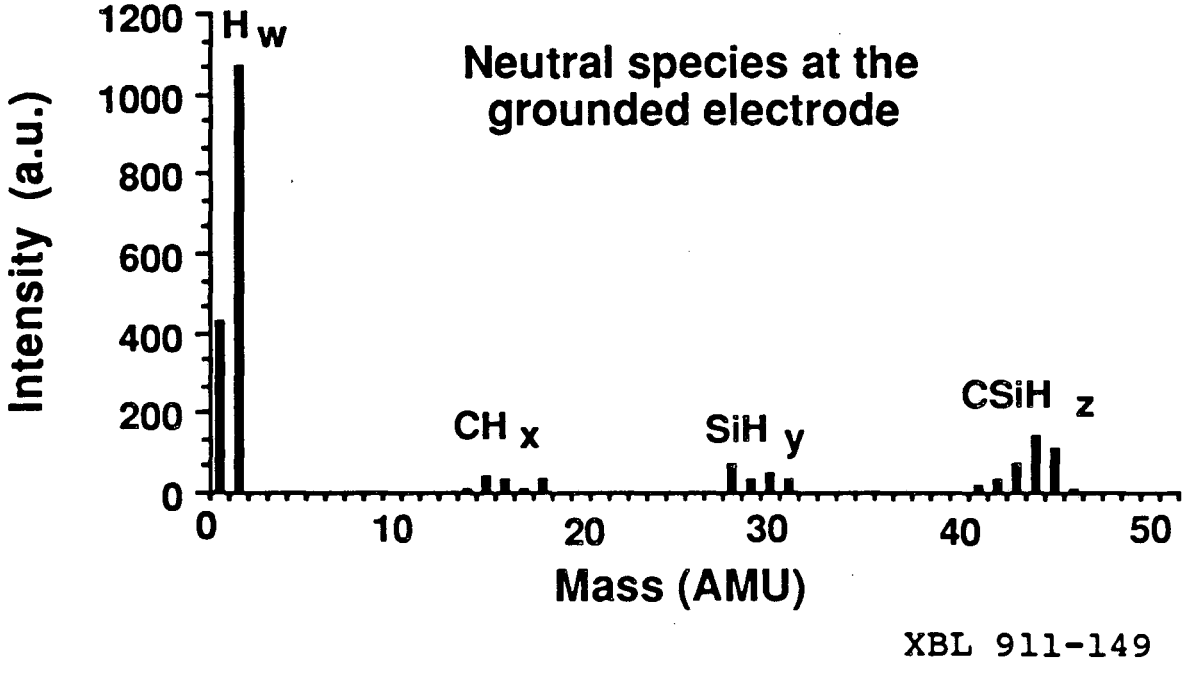

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Fig. 1

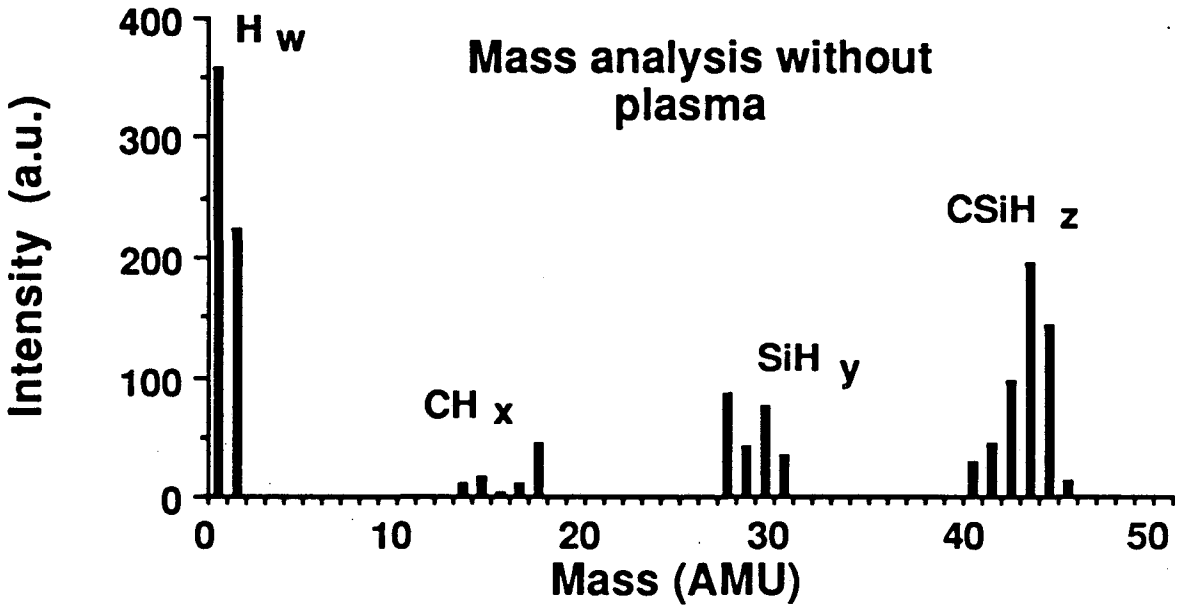

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Fig. 2

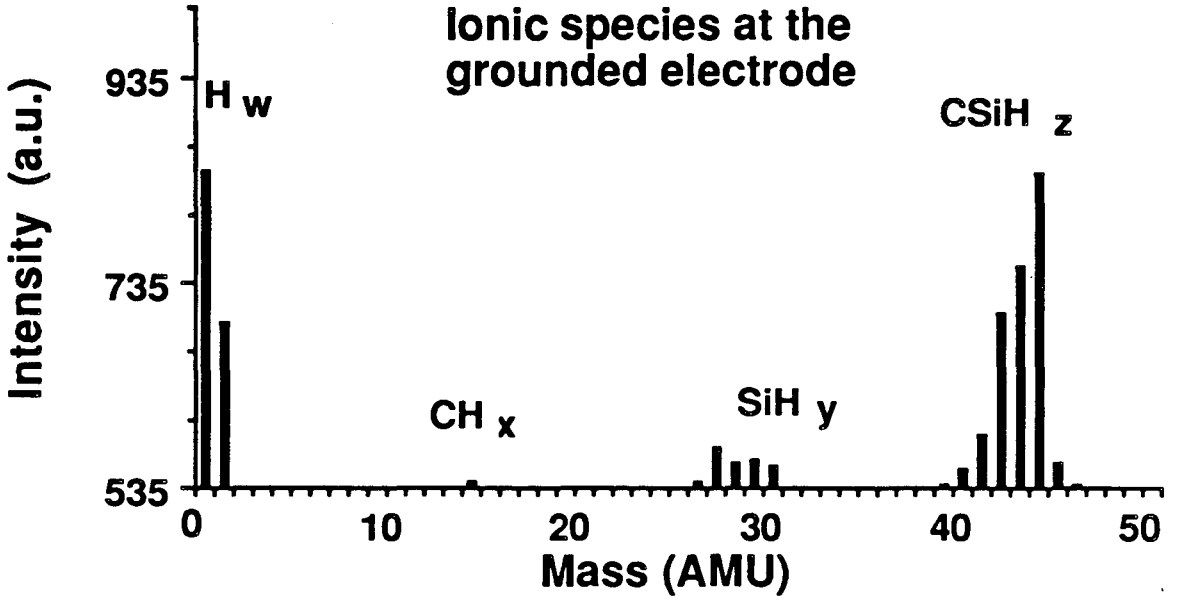

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Fig. 3a

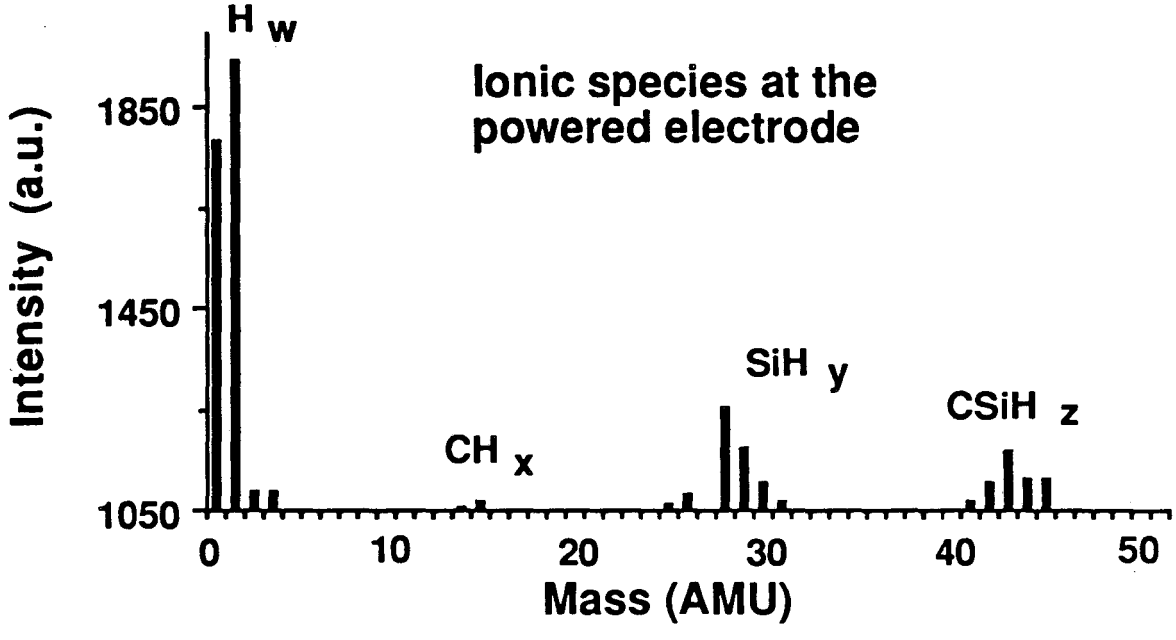

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Fig. 3b

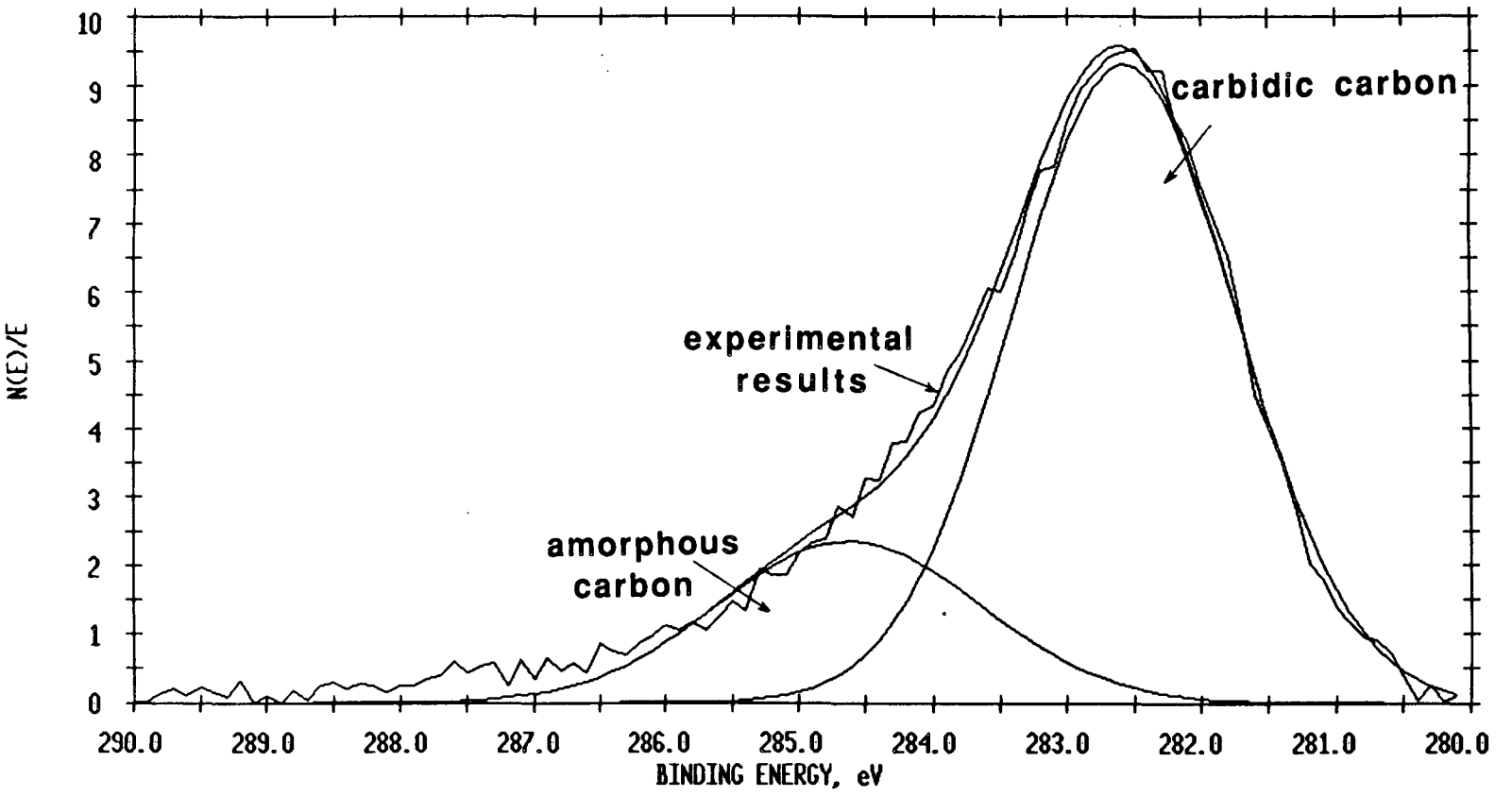

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Fig. 4

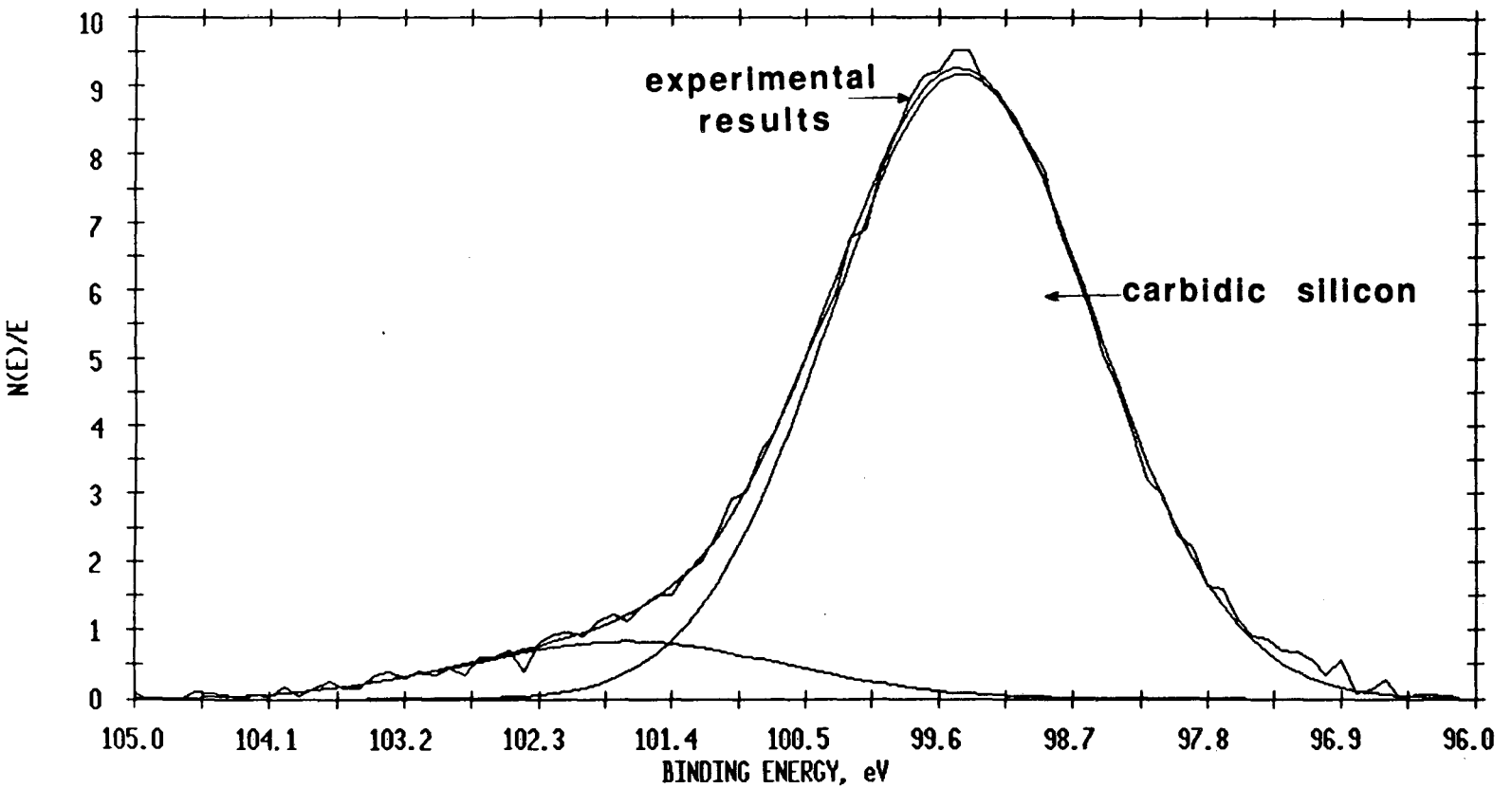

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Fig. 5

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