LANGMUIR PROBE AND ENERGY-MASS SPECTROSCOPY MEASUREMENTS IN ECWR PLASMAS FOR DIAMOND DEPOSITION

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Abstract

An RF experiment was set up with a static magnetic field perpendicular to the high frequency magnetic field. The reactive plasmas consist of variable concentrations of methane in hydrogen with small admixtures of oxygen. The conditions were chosen for diamond deposition on Si-substrates heated by an electrical current to 1120 K. During the deposition processes, the plasmas were investigated by means of an energy and mass spectrometer (EMS) and an automatic Langmuir probe. The retractable probe wire is connected to a fast and high resolving electronic measuring equipment and is controlled by a personal computer.

Introduction

Large-area plasma enhanced deposition of different carbon coatings requires a uniform substrate temperature and a homogenous plasma at the front of the whole substrate. An RF experiment was set up with an inductive and/or capacitive coupling and a directed magnetic field perpendicular to the high frequency magnetic field.

During the deposition process in various methane-hydrogen and methane-hydrogen-oxygen mixtures at different concentrations, the plasma was investigated by means of an energy and mass spectrometer (EMS) and an automatic Langmuir probe. The probe measurements provided the electron densities, the electron energy distribution functions, the mean electron energies (electron temperatures) and the plasma potentials as functions of the concentrations of the various mixtures and the total pressure.

The EMS measurements provided the neutral particle density and the ion energy distribution as functions of the above mentioned parameters. The plasma potential of the probe measurements were compared with the results of the energy and mass spectroscopy. The agreement of these independent measurements is very satisfactory, thus confirming the automatic probe measurements.

Experimental setup and diagnostics

The plasma reactor (Fig. 1) [1] was built of a stainless steel chamber containing the normally grounded substrate electrode (molybdenum), a load-locked substrate exchange unit L, a Langmuir probe LP and an energy and mass spectrum analyzer A. The upper part of the reactor contains a quartz vessel Q (Ø 20 cm), an upper electrode plate E (aluminum) and a gas feed system. The latter is separated in a gas inlet through the electrode E and a lower gas injection ring R. This increases the deposition yield on the substrates and avoids excessive deposition in the quartz vessel. The plasma is driven by a one-turn coil C at 27 MHz and superposed by a homogeneous magnetic DC field. The refractive index of the plasma, which is strongly influenced by the stationary field, was adjusted in such a way as to build up a stationary wave in the reactor. This electron cyclotron wave resonance (ECWR) [2] occurs at pressure values up to some Pa. In this kind of plasma reactor the skin effect could nearly be prevented. Therefore it had a large area of almost constant electron density over the cross section of the reactor. Pumping was done by turbo pumps (base reactor pressure $5*10^{-8}$ mbar, leak rate 10^{-5} mbar l/s). The influence of N_2 and other impurities during the deposition was very low, the main impurities in the residual gas spectrum was H_2 (traces of H_2O, CO_2 and CO).
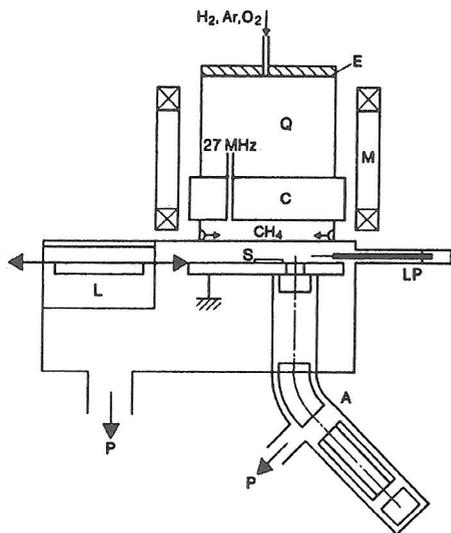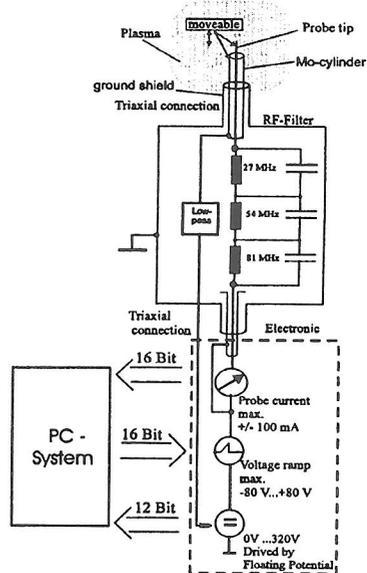

Fig. 1: ECWR reactor setup

Fig. 2: Langmuir-probe setup

To change the substrate bias independently of the RF power in a wide range, we used this reactor, which combines the advantages of both inductive and capacitive excitation. The RF-power from 1 to 700 W was coupled in by an one-turn coil, which was grounded on one side. In order to suppress an asymmetrical capacitive coupling, caused by the voltage drop along the coil, a slotted aluminum cylinder was mounted

inside the vessel. By varying the coupling of the polarizer against ground potential, a cylindrically symmetric discharge from pure inductive ($V_{pl} \approx 20$ V) to capacitive ($V_{pl} \approx 100$ V) could be obtained. Further on the substrate holder is connected to a variable bias voltage which could be chosen from -300V to 0 V. During diamond deposition the Si-substrate is electrically heated. The substrate temperature is controlled by a single chip microprocessor and calibrated by means of an optical pyrometer. For nucleation a substrate bias voltage of about -300 V is used to increase the nucleation density [3]. The pressure is controlled by two baratron vacuum gauges and ranged from 0.2 Pa to 20 Pa. The total flux was kept constant at 50 sccm by means of a regulated throttling valve and calibrated flow controllers.

The Langmuir probe measurements could be carried out by means of a 100 µm W-wire probe tip with 10 mm length. The compensation electrode consisting of a 6 mm Mo-cylinder was positioned just behind the probe tip (Fig. 2). The probe tip, as well as the Mo-cylinder was retracted in separate capillary tubes of quartz glass during the measuring breaks. The electronic equipment (Fig. 2) consists of a computer controlled automatic measuring system, which generates a voltage ramp from -80 V to +80V. The probe current could be measured in 8 different ranges from 31.6 µA to 100 mA full scale and converted in 16 bit. The compensation electrode is connected with an imped-ance transformer (1:1) to provide a low resistance floating potential as the basis for the voltage ramp. The measuring time of one I-V-pair takes only 19.5 µs (1024 values per 20 ms). In this way it was guaranteed, that the probe tip was exposed to the plasma only for a short time. Coatings that still occur after long time of measuring period could optionally be sputtered off by a negative voltage or be evaporated by positive voltage.

In order to minimize the described capacitive coupling [4] of the feed line of the probe, it was completely built up in triaxial technique. The compensation electrode was extended as an interior shielding. Its coupling to the plasma with about 100 pF is much stronger than the coupling of the probe itself (< 1 pF). The capacitance between the interior shielding and the grounded outer screen is 4.5 pF, which is also much smaller than the coupling to the plasma. Because of the 16 pF coupling between the feed line of the probe (inner conductor) and the feed line of the compensation, an extra coupling capacitance is not needed.

Before the total automatic evaluation [5] took place the I-V-curve was smoothed in the frequency range (see Fig. 3c) using filter functions, like Gauss, Blackman, Hanning, Hamming.

The results of the evaluations are shown in a 4-figure-diagram including the most important plasma parameters. Figure 3a shows the I-V-characteristic of a ca-pacitively excited methane-hydrogen-oxygen plasma with 500 W RF-power input at a pressure of 1 Pa. The average electron energy $<E_e>$ was calculated with three different methods. The slope of the logarithmic electron repelling current is especially reliable in the case of a high plasma density which is related with a Maxwell-distribution exhibit-ing low deficiency in the high energy domain. The slope of the second derivative of the electron repelling current in a logarithmic plot (figure 3b) shows its advantages in the case of low plasma density. This often leads to a larger deviation from the Maxwell

distribution and a higher average electron energy. The integral method is to be preferred in the case of all non-Maxwell distributions. The electron density n_e is also calculated in three ways: by evaluation of the electron saturation current, by measuring the current at the plasma potential and by integrating the electron repelling current. Those three methods show corresponding results with a deviation of ±10%. In figure 3d the second derivative of the probe current I"(V) is shown over the whole measuring range. The slope indicates that the compensation with the triaxial-lead is working in spite of a large RF-modulation of plasma potential. The frequency spectrum is shown in figure 3c. Disturbances may be seen at 50 Hz, 100 Hz and so on. They are caused by the DC-power supply of the RF-generator not properly filtered.

After four months of continuous use in coating CH_4 plasmas with about 100 measurements per day no influences on the retractable probe tip could be detected.
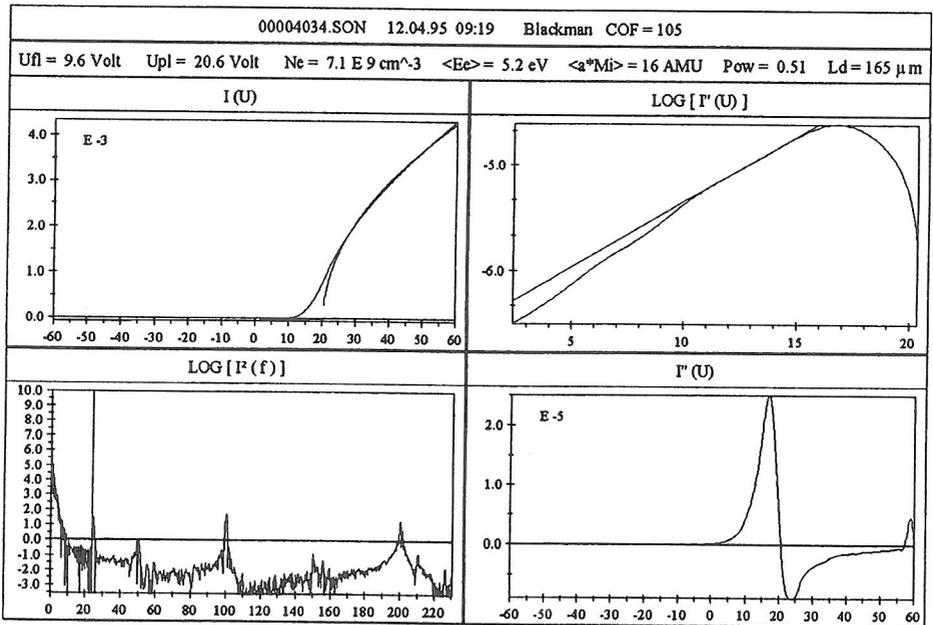

Fig. 3a,b,c,d: Plasma parameters and graphical evaluation of one Langmuir-probe measurement

Results and discussion

In Fig. 4 a comparison of the plasma potential Vp determined by two independent methods for a methane-hydrogen-oxygen plasma is shown. The carbon to oxygen content was in the ratio of 1:1, both fluxes were 10% of the total flux. Vp was derived from probe measurements and energy and mass spectroscopy. The pressure variation in this plasma provided good agreement at low pressure values between the maximum of

the ion energy distribution and Vp. This is due to the model that all ions are collected from a surface which is on the same potential as the plasma, without any collisions on their way to the surface. At higher pressure values the agreement of the maximum ion energy and Vp is rather good, because the ions are loosing energy on their way through the sheath as a result of collisions. The decrease of the plasma potential indicates the decrease of the electron temperature with increasing pressure (see also Fig. 6).
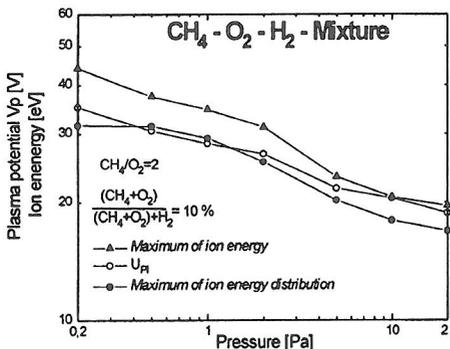

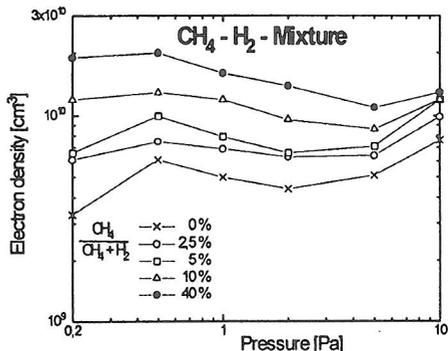Fig. 4: Plasma potential Vp measured with a Langmuir probe and an energy-mass spectrometer

Fig. 5: Electron density for various methane concentrations in hydrogen measured with a Langmuir probe

In Fig. 5 the electron density of a methane hydrogen plasma without oxygen is plotted as a function of the total pressure. The various methane concentrations ranged from 0% to 40%. At the lowest pressure values from 0.2 Pa to 2 Pa a balance between the particle loss by ambipolar diffusion and a gain by electron collisions may be assumed. For this reason the smallest methane concentrations show the lowest electron densities, because the diffusion coefficient of hydrogen is considerably larger than that of all other molecules or radicals in this plasma. Especially at 0.2 Pa this effect could be observed. With increasing pressure the different concentrations converge. This behavior is due to an increasing volume recombination. At 20 Pa the wall recombination may be of the same order of magnitude as the volume recombination. This means that the electron density is not as much influenced by the various diffusion coefficients of the different concentrations as at 0.2 Pa. However all methane concentrations show the same pressure dependence with a maximum at the ECWR resonance at 0.5 Pa. This is due to the prevented skin effect in the near of the wall and the increase of the electron density in the middle axis of the reactor. The slight drop with increasing pressure between 0.5 and about 5 Pa is due to an increase of the damping of the resonance, which does not occur at higher pressure values. For higher values than 5 Pa the increasing influence of electron generation by an increasing collision frequency between heavy particles and electrons exceeds the damping of the resonance. The behavior of the mean electron energies in the same methane hydrogen concentrations as before are shown in Fig. 6. The mean energies also depend on the diffusion coefficients of the various mixtures,

i. e. the largest diffusion coefficients are related to the highest losses of charged particles. From there the mean electron energy, in the case of Maxwellian energy distribution the electron temperature, is a steep function of the total pressure.
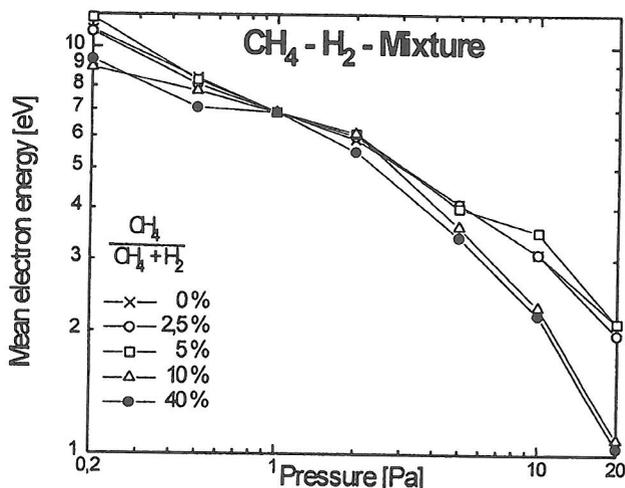

Fig. 6: Mean electron energy of various methane concentration measured with a Langmuir probe

Conclusions

It was the purpose of the present investigations to measure in situ the parameters of reactive plasmas under diamond deposition conditions by means of a powerful tool for technological plasmas, the automatic Langmuir probe. To ensure the probe data, the plasma potential was derived from independent EMS-measurements of the ion energy distributions at several concentrations. Because low pressure plasmas are far from thermal equilibrium, it is necessary to have reliable data of the mean electron energy and electron density to calculate the molecule, ion and radical densities in the different plasmas. Without these parameters no modeling should be able, since the electron collisions as the most important processes are steep functions of the aforementioned parameters. Ultimately an upscaling of reactors and a comparison of different concepts should only be possible if the complicated plasma kinetics could be understood on the basis of these parameters.

References

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