CHARACTERIZATION OF OXYGEN/TEOS PLASMAS IN A HELICON DIFFUSION REACTOR USING MASS SPECTROMETRY AND OPTICAL EMISSION SPECTROSCOPY

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ABSTRACT

We develop a low pressure (1-10 mTorr) RF helicon reactor in order to deposit thin films of SiO_2 in its diffusion chamber from O_2/TEOS plasmas. To optimize the density of oxygen atoms, the plasmas in O_2 and O_2-N_2 mixtures are investigated using langmuir probes, emission spectroscopy and mass spectrometry. Oxygen atoms are successfully measured by actinometry and ionization threshold mass spectrometry. The O atom fraction increases with the injected power. It is estimated to 10% in O_2 plasmas (P=400W) and increases to 20 % in a 95%O_2-5% N_2 mixture.

INTRODUCTION

The Helicon source is one of the new generation of "high density, low pressure" reactors that are of interest for microelectronic applications. We use one as an atomic oxygen source to enhance the deposition of SiO_2 from TEOS ($Si(OC_2H_5)_4$), which can be preferred to silane since it is non toxic and yields better step coverage. Since high densities of oxygen atoms are required for deposition of SiO_2 films, our aim was first to measure and optimize the density of oxygen atoms in an oxygen helicon plasma, which is presented thereafter.

EXPERIMENTAL SETUP

The helicon reactor is schematically shown in Fig 1. The plasma source is a glass tube, 15 cm in diameter and 30 cm in height surrounded by an Helicon antenna [1] and a solenoid creating a static magnetic field inside the plasma. The plasma source is operated in the pressure range 1-10 mTorr with RF powers (13.56 MHz) up to 700 W. The plasma diffuses into the diffusion stainless-steel chamber which is 30 cm in diameter and height. The applied magnetic field is typically 60 Gauss in the center of the source

(B_0). Since there is no solenoid around the diffusion chamber, B is very weak at this level (10-20 Gauss). The reactor is pumped by a turbo/rotary pumping device and the base pressure is 2 10^{-6} Torr. The pressure is regulated owing to an exhaust slide valve and oxygen flowrates up to 80 sccm can be injected at 10 mTorr.

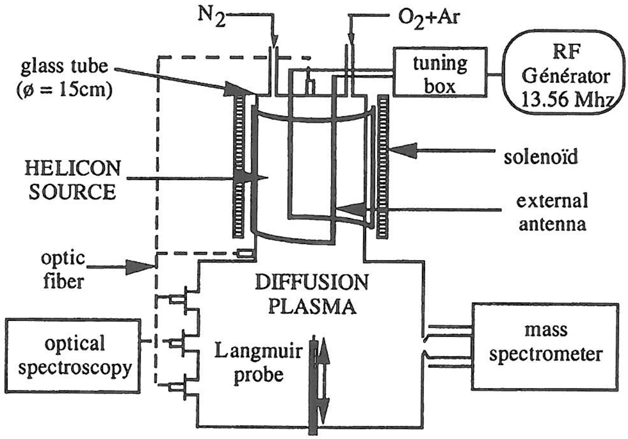

Fig 1 : Experimental setup

DIAGNOSTICS

Three kinds of diagnostics are used to characterize the plasma obtained in the diffusion chamber : langmuir probes, actinometry and mass spectrometry.

Langmuir probes : a cylindrical probe (ϕ = 0.1 mm) can be moved along the axis of the diffusion chamber. The probe curves are recorded without any compensation of RF interferences. They are analysed with conventional theories with the following assumptions : the energy electron distribution function (eedf) is maxwellian, the effect of B is neglected, the positive ion is O_2^+, the contribution of O^- to the electronic current is neglected. Thus we only expect to have rough estimations of the ion density, the electron temperature (T_e), the floating potential (V_f) and plasma potential (V_p).

Actinometry : in order to follow the oxygen atom density we added 5% argon and recorded the actinometry signal I_O(844nm)/I_{Ar}(750nm) at different positions on the diffusion chamber and on the axis of the reactor (cf. Fig 1). The light emitted by the plasma is collimated on an optical fiber and analysed by a monochromator JY 320. The response of the optical system with the wavelength was determined in relative values using a tungsten lamp.

Mass spectrometry : sampling of neutral and ionic species is achieved with a Balzers PPM 421 plasma monitor, which is a differentially pumped mass spectrometer with an energy filter (cylindrical mirrors analyzer) between the ion source and the quadrupole mass filter. The ion flux is measured by a secondary electron multiplier. The sampling aperture (100 μm in diameter) is close to the wall of the diffusion chamber (cf. Fig 1). The base pressure inside the PPM is 5 10^{-8} mTorr.

The ion energy distribution function of positive ions (cf. Fig 2) escaping from an oxygen plasma (O^+ and O_2^+) show a peak at an energy equal to the voltage drop on the sheath at the wall, i.e. between the plasma potential and the earthed analyser. Thus the ion energy in the mass spectrometer is expected to be a measure of V_p.

For the analysis of neutral species, ions coming from the plasma are repelled by positive biasing of the entrance optics. The ionization chamber is located 4 cm away from the sampling hole. The energy of the electrons in the ionization chamber can be varied from 8 eV (with an estimated energy spread of about 0.5 eV). Oxygen atoms are analysed using threshold ionization technique, as previously developed by Sugai et al [2] and Kae-Nune et al [3] in the case of CH_x and SiH_x radicals. The intensity at mass 16 (O^+) is recorded as a function of the electron energy with and without plasma. Since the threshold of the dissociative ionization of O_2 is 19.5 eV while the threshold of direct ionization of O is only 13.6 eV, the increase in intensity at mass 16, observed in the electron energy range 13.6-19.5 eV, is proportional to the atom density. An example of experimental variations of I(16 amu) versus the electron energy is shown in Fig 3. Below 19.5 eV, the background signal (at 16 amu) is zero. The existence of a small signal when the plasma is off is attributed to a two-step process in the ionization chamber (O_2+e-->O+O+e followed by O+e--> O^++2e).

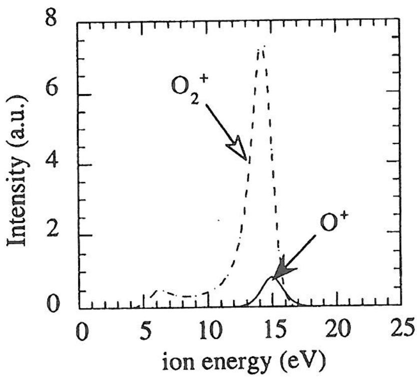

Fig 2 : Ion enegy distribution function under conditions (A), 95%O_2-5%N_2, P = 200 W

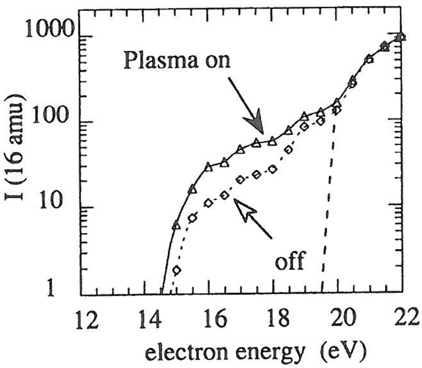

Fig 3 : Intensity at 16 amu versus electron energy; conditions (A), 95%O_2-5%N_2, 400 W

RESULTS IN O_2 AND O_2/N_2 PLASMAS

All the results presented afterwards were obtained at mid height of the diffusion chamber but at different radial positions : on the axis for probe curves, close to the wall for mass spectrometry and actinometry are radially averaged. The usual experimental conditions are (A) : 10mTorr, 50 sccm, B_0=60 G and (B) : 1 mTorr, 8 sccm, B_0=60 G. Results concerning positive ions and plasma potential are presented followed by results on the O atom density in the helicon reactor.

Positive ions and plasma potential

Some results deduced from probe curves in oxygen plasmas were previously presented [4] and, here, only the main results are reported : the ion density increases with the RF power, typically from 10^{10} cm^{-3} at 50 W to 5 10^{10} cm^{-3} at 500 W. Measured values of T_e are almost independent of the RF power at fixed pressure : T_e is about 6 eV at 1 mTorr and 3 eV at 10 mTorr.

As expected and previously shown by C. Charles [5], O^+ and O_2^+ are observed by ion mass spectrometry. The ratio of the intensities of O^+ and O_2^+ peaks ($I_{O^+} / I_{O_2^+}$) increases with the RF power, but O_2^+ is always the main positive ion. The increase in O^+ fraction with the power is attributed to the increase of [O] (cf. below). The variations of V_p (deduced from the probe curves) with RF power are plotted in Fig 4 and are compared to values of V_p measured by ion mass spectrometry. Both measures show a decrease in V_p occuring at the transition between capacitive and inductive modes [1]. Both measures also exhibit a decrease in V_p as the pressure is increased, which means that the energy of ions impinging on a grounded wafer will be higher at low pressures. Nevertheless, discrepancies are observed between both measures of V_p, particularly at 1 mTorr. This discrepancy is perhaps due to the fact that, according to the diagnostic, V_p is measured either on the axis (probe) or close to the wall (mass spectrometry).

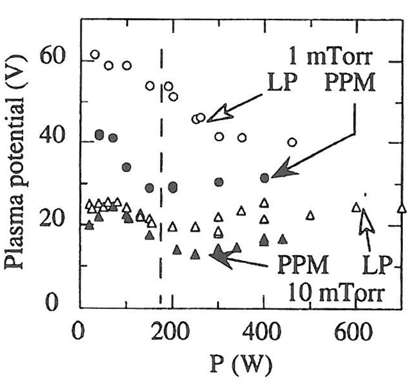

Fig 4 : Variations of Vp with the RF power, conditions (A) and (B), O_2 plasmas

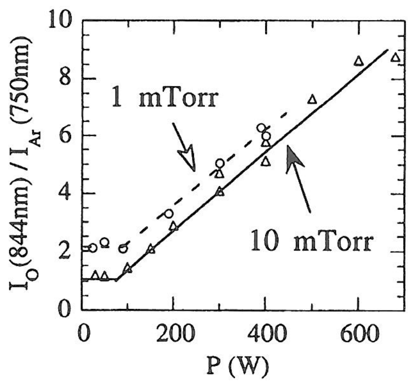

Fig 5 : Variations of the actinometry signal with the power, conditions (A) and (B), O_2 plasmas

Density of oxygen atoms in O_2 and O_2-N_2 plasmas

For several years, the question of whether actinometry is valid or not to monitor oxygen atoms has been largely discussed. Typical evolutions of I_O/I_{Ar} with the RF power obtained in the helicon reactor at 1 and 10 mTorr are plotted in Fig 5. These experimental curves $I_O/I_{Ar}(P)$ were previously compared to computed values of I_O/I_{Ar} at the corresponding electron temperatures (cf. details in [6]). It was concluded that the limit values obtained at low powers (below 100 W) corresponded to the creation of O* by dissociative excitation of O_2. Thus actinometry is only valid at high RF powers provided the dissociative excitation contribution is substracted. Furthermore, an estimation of the atom fraction [O]/N (where N denotes the total neutral density, $N=p/kT_{gaz}$) can be given : about 15 % atoms in a 10 mTorr oxygen plasma at 700 W.

Further proofs of the validity of actinometry at high RF powers are now given by ionization mass spectrometry on O atoms in O_2 and O_2/N_2 plasmas. The difference between the I^{16}_{on} and I^{16}_{off}, denoted ΔI, integrated in the energy range 15-18 eV is expected to be proportional to [O]. In the case of CH_x and SiH_x radicals [2-4], absolute concentrations of radicals (in the vicinity of the extractor) could be deduced from ΔI, assuming that their loss on the extractor wall was negligible. Because of the strong reactivity of O atoms on metallic surfaces, it is likely that their recombination can not be neglected. Thus, only relative values are presented thereafter.

Fig 6 shows the variations of ΔI with the RF power in a 10 mTorr 95% O_2 - 5% N_2 mixture. The O atom fraction, estimated from actinometry under the same experimental conditions, are reported too. Thus, it is shown that actinometry and ionization threshold mass spectrometry agree very well. The results presented above were obtained in 95% O_2 - 5% N_2 mixture since the O atom density is much higher than in a pure oxygen plasma. Such a behaviour is consistent with the literature since similar trends were previously reported in microwave surface discharges [6], in DECR plasmas [7] and in the positive column of DC discharges [8]. Both actinometry and ionization threshold mass spectrometry were used to follow the O atom fraction versus the N_2 percentage in the O_2/N_2 mixture. Fig 7 shows the increase in [O], which is clearly demonstrated by both diagnostics. These experiments where very instructives : first, the fact that the oxygen dissociation degree can be increased by adding N_2 can be interesting for processes requiring a high density O atoms; second, it enabled to confirm the validity of actinometry to follow [O] in the diffusion chamber of a helicon reactor. Nevertheless, the basic question of the mechanisms responsible for this increase in [O] is still open, although the most likely explanation is a decrease in the wall recombination frequency of O in the presence of nitrogen [6-7].

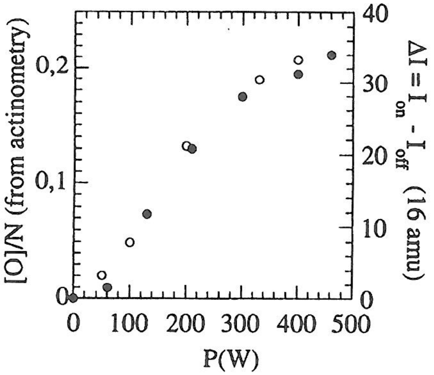

Fig 6 : Variations of [O]/N (from actinometry) and ΔI(16 amu) (mass spectrometry) versus power, conditions (A) 95% O_2-5%N_2

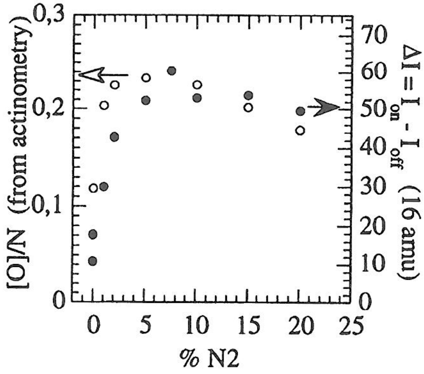

Fig 7 : Variations of [O]/N (from actinometry) and ΔI(16 amu) (mass spectrometry) versus N_2 percentage, conditions (A) 400 W

CONCLUSION

This study of oxygen plasmas created in a helicon reactor, preliminary to deposition in O_2/TEOS mixtures, yielded interesting results, particularly on oxygen atoms. First, it was shown that their density could be followed by ionization threshold mass spectrometry. Second, it was shown that, except at low power, actinometry could be used to monitor O density. At 1 and 10 mTorr, the atom fraction increases with the power, reaching about 15 % at 700 W, values much larger than those recently measured by K. Kadota in a high power pulsed helicon plasma [9]. Furthermore it was shown that [O] was roughly doubled when five percent nitrogen was added to the oxygen gas feed.

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