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Optical emission spectroscopy investigation of sputtering discharge used for SiO_xN_y thin films deposition and correlation with the film composition

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Abstract

The r.f. discharge of sputtering silicon target using argon–oxygen–nitrogen plasma was investigated by optical emission spectroscopy. Electronic temperature (T_e) and emission line intensity were measured for different plasma parameters: pressure (from 0.3 to 0.7 Pa), power density (0.6–5.7 W cm^{-2}) and gas composition. At high oxygen concentration in the plasma, both T_e and the target self-bias voltage (V_b) steeply decrease. Such behaviour traduces the target poisoning phenomenon. In order to control the deposition process, emission line intensity of different species present in the plasma were compared to the ArI (λ = 696.54 nm) line intensity and then correlated to the film composition analysed by Rutherford Backscattering Spectroscopy.
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1. Introduction

Silicon oxynitride (SiO_xN_y) thin films are very interesting materials due to the possibility of tailoring their optical and electrical properties by changing the stoichiometry of the deposit. Hence, they can be employed for passivation and masking in microelectronic device technology, as antireflective layers on selectively solar-absorbing surfaces or for luminescent devices [1–3]. These materials have been deposited on various substrates with several methods such as chemical vapour deposition and reactive magnetron sputtering [4–7]. In this work, SiO_xN_y thin films were deposited by reactive magnetron sputtering using a silicon target and different $Ar:O_2:N_2$ atmospheres. As the optical and electrical properties of the films depend on the deposition conditions, it becomes necessary to control the deposition process. In the case of a reactive sputtering process, the major disadvantage is due to the target poisoning. Optical emission spectroscopy is a powerful tool to study the composition of the sputtering atmosphere and other deposition parameters such as the plasma electronic temperature. Moreover, the variation of the target self-bias voltage provides information about the evolution of the target state under different sputtering conditions. In this paper, the variation of the target voltage was followed and a correlation between the process parameters and the relative intensity of emitting species was established. Then, the electron temperature dependence on gas pressure and composition by using silicon and argon emission line intensities was studied. These results permitted to link the relative intensities emitted by some plasma species to the thin film composition.

2. Experimental

Silicon oxynitride films were deposited in a r.f. sputtering unit (Alcatel SCM 450) from a pure silicon target (100 mm diameter, 99.999% purity). Different power densities (from

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0.63 to 5.73 W cm^{-2}), argon–oxygen–nitrogen atmospheres and total pressures (<1 Pa) were used. Before deposition, the sputtering chamber was evacuated to 10^{-4} Pa and the target is pre-sputtered in an argon atmosphere during 30 min to eliminate the surface pollution. Whatever the pressure, the argon concentration in the gas mixture was 70%. The ratio $x = [O_2]/([O_2] + [N_2])$ was varied from 0.1 to 0.5 (where $[O_2]$ and $[N_2]$ are the oxygen and nitrogen flow rates, respectively). Optical emission spectroscopy (OES) investigations were performed using a Jobin-Yvon 270M spectrometer. The light was collected using an optical fibre situated outside a quartz window of the sputtering chamber. The spectra were recorded in the range of 250–850 nm and the spectral response of the detector was calibrated using a reference standard lamp. The deposition rate was deduced from the film thickness (measured by profilometry) and the deposition time. The elemental analysis of the deposited films was investigated by Rutherford Backscattering Spectrometry (RBS) at CERI-CNRS (Orléans, France), using 2 MeV alpha particles and a 15 nA current intensity. Vitreous carbon substrates were used so that the RBS signal of the film components appeared at higher energies than the substrate element and did not overlap. In order to get the concentrations of all relevant elements (Si, O, N), the experimental spectra were simulated with SIMNRA program.

3. Results and discussion

Characteristic emission lines were observed for the different species present in the plasma. The great number of intense lines for argon emission made difficult the observation of the lines of other elements. Nevertheless, we distinguished at least one line per element present in the plasma. The most intense ones were chosen; ArI at 696.54, 703.02, and 706.72 nm, SiI at 251.43 nm, OI at 844.63 nm and N_2I at 337.13 nm.

Using these lines, one can access to different plasma parameters and have a better understanding of the deposition process. Indeed, for neutral species and assuming a direct process for electronic excitation, the signal intensity detected at frequency corresponding to the transition p → q, is given in Ref. [8]:

$$I_X = [X_p^*] R_{pq} h\nu_{pq} A_{pq}\, d\Omega'$$

where, $d\Omega'$ is the solid angle inside which the light is collected from the plasma by the optical fibre; its value remains constant. A_{pq} is the transition probability, $[X_p^*]$ the density of the element X at the excited state “p” inside the discharge, R_{pq} the spectral response of the apparatus and h is the Planck’s constant.

On the other hand and for plasma considered in the partial local thermodynamic equilibrium, one can determine the electronic plasma temperature using the relative intensity between two spectral lines corresponding to the transition of the different lower levels according to the following expression [9]:

$$T_e = \frac{(hc/k)(E_m - E_p)}{\ln\left[\frac{I_{pn} A_{ms} g_m \nu_{ms}}{I_{ms} A_{pn} g_p \nu_{pn}}\right]}$$

where E_m and E_p are the energies of levels, g_m and g_p are the statical weights. k is the Boltzmann constant and c is the speed of the light.

In order to minimize the error in the calculation of the electron temperature, the difference between energy levels should be large and wavelengths of selected lines should be neighbouring. The different parameters of the Ar lines used for T_e calculation are taken from NIST database [10].

Before studying a reactive complex system like $Ar{:}O_2{:}N_2$, the influence of the pure Ar plasma pressure and power density on the emission lines intensity and electronic temperature was investigated. As shown in Fig. 1, when the argon flow rate increases at a fixed power density (here, 3.18 W cm^{-2}), the intensity of the argon and silicon emission lines and the electron temperature decrease. This behaviour is the consequence of the increase of the collision frequency of electrons with neutral argon atoms due to the pressure increase, which leads to a decrease of the average electron energy [8,9]. Moreover, an increase in discharge power at fixed Ar flow rate (here, 5.5 sccm) leads to an increase of emission line intensity and a decrease of electron temperature values (Fig. 2). Clenet et al. [8] reported an increase of the electron density and line intensity with power. However, the expression of electron density versus Ar line intensity shows that electronic temperature is inversely proportional to electron density; this is in agreement with our results.

As said before, the main goal of this work is the use of silicon target to deposit silicon oxynitride films. So, it was interesting to examine the influence of the reactive atmosphere on the process parameters. Fig. 3a shows the variation of electron temperature determined for different plasmas under a fixed discharge power (here, 3.18 W cm^{-2}) and different total pressures. T_e remains almost constant for 0.3 Pa, whereas, at

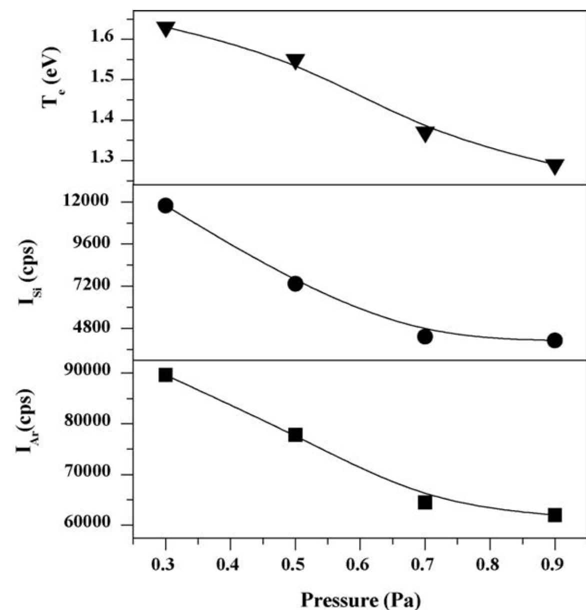

Fig. 1. Variation of the ArI (λ = 696.54 nm) and SiI (λ = 251.43 nm) line intensities and the electronic temperature with pressure in the case of a pure argon plasma.

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