IN SITU MONITORING OF PLASMA ETCHING AND DEPOSITION BY USING THE GAS PRESSURE DIFFERENCE

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The gas pressure variation due to the chemical reactions in discharge plasma is analysed using flow and mass balance equations. A good correlation was found between behaviour of the gas pressure drop and the etching rate of aluminium. Theoretical and experimental analysis has shown that the gas pressure variation could be used as an efficient tool for *in situ* monitoring of the plasma etching and/or deposition.

Origin of the gas pressure difference in the plasma reactor

There is a number of processes which influence the gas pressure in the plasma chemical reactor. In this contribution the effect of the chemical reactions on the total gas flow and conductance of vacuum system is discussed. In a gas mixture the partial pressure of j-th component p_j can be formally written down as

$$p_j = Z_j Q_j \,, \tag{1}$$

where Q_j is the mass flow rate and Z_j is vacuum resistance defined as

$$Z_j = \frac{1}{S_j} = \frac{1}{S_n} + \frac{1}{C_j} \,. \tag{2}$$

Here S_j, S_n are the effective and nominal pumping speed, C_j is the conductance of the vacuum duct between the pump and plasma reactor. Let Z_{jo}, Q_{jo} and Z_j, Q_j denote the vacuum resistance and mass flow rate in the states "plasma off" and "plasma on", respectively. Then the change of total gas pressure due to occuring reactions is

$$\Delta p = p - p_o = \sum_j (Z_j Q_j - Z_{jo} Q_{jo}) \,. \tag{3}$$

In the case when one overall chemical reaction (in the gas phase or at treated surface) prevails, the related gas flow rate difference $\Delta Q_j = Q_j - Q_{jo}$ can be expressed by means of the reaction rate $\mathcal{R}$ (in s^{-1})

$$\Delta Q_j = kTa_j\mathcal{R} \,, \tag{4}$$

where k, T, a_j are the Boltzmann constant, the gas temperature and j-th component stoichiometric coefficient, respectively. The influence of the variation in the flow rates on the total gas pressure depends on the character of the gas flow.

In the case of molecular flow regime, the value of Z_j is independent on the gas mixture composition and so $Z_j = Z_{jo}$. Thus, the relation (3) becomes

$$\Delta p = \sum_j Z_{jo} \Delta Q_j = kT\mathcal{R} \sum_j Z_j a_j \,. \tag{5}$$

According to (5), the gas pressure difference is proportional to the reaction rate. The relation (5) can be used to determine the total reaction rate (of etching or deposition) from measured values of Δp, when stoichiometry and Z_j are known. In the case of viscous flow, the vacuum resistance is the same for all species, i.e. $Z_j = Z$. The initial value $Z = Z_o$ is changed due to changing of the relative composition of the mixture when the discharge is excited. When introducing the total flow rates $Q_o = \sum_j Q_{jo}$ and $Q = \sum_j Q_j$ then Eq.(3) gives

$$\Delta p = ZQ - Z_o Q_o = (Z - Z_o)Q_o + Z\Delta Q \,, \tag{6}$$

where the total flow rate difference $\Delta Q = Q - Q_o$ was introduced. In this equation, the first term represents a contribution caused by the change in the vacuum resistance and the second one expresses a change of the pressure due to the gas flow rate difference.

In the viscous flow regime, the vacuum resistance depends on the total gas flow rate and the gas mixture composition, too. The influence of these two effects can be investigated experimentally by measuring the flow characteristics $p = p(Q)$ in various mixtures. If the effect of the gas mixture composition on vacuum resistance is negligible then only an influence of the total flow rate should be taken into consideration. Then the flow rate difference ΔQ due to prevailing chemical reaction can be determined as a difference of gas flow corresponding to the values p and p_o using $p - Q$ characteristic

$$\Delta Q = Q(p) - Q(p_o) \,. \tag{7}$$

Pressure variation during etching of aluminium

To avoid complications about comparing the theory with experiment, the etching of aluminium by chlorine was chosen. This process has the advantage that after removing of the native oxide layer in the $SiCl_4 + Cl_2$ plasma, the etching continues spontaneously when no RF discharge is present. Such a situation simplifies the conditions during the etching process as the concentration of reactive species — molecular chlorine — is known.

The etching of aluminium layer at the low temperature can be described by the reaction

$$\mathrm{Al} + \frac{3}{2}\mathrm{Cl_2} \longrightarrow \frac{1}{2}\mathrm{Al_2Cl_6} \tag{8}$$

or

$$\mathrm{Al} + \frac{3}{2}\mathrm{Cl_2} \longrightarrow \mathrm{AlCl_3}\,. \tag{9}$$

The sum of stoichiometric coefficients of all gas species u is equal to -1 for (8) and -0.5 for (9). In all cases when $u \neq 0$ a difference occurs between the flux of the active gas reaching the surface and the flux of the etching product releasing the surface so that a change in the pressure can be observed in the etching chamber during etching [1].

The atom flux of an etched material I_o and the etching rate R can be related by

$$I_o = \frac{A\rho R}{m}\,, \tag{10}$$

where A is the area of the etched layer surface, ρ and m are the density and mass of the atoms of etched layer. Then the flux of the active (chlorine) particles which strike the etched surface can be calculated as $\frac{3}{2}I_o$. The flux of etching products leaving the surface is given by $\frac{1}{2}I_o$ for (8) and I_o for (9). As a result the flux of all molecules pumped from the chamber differs from the flux of all molecules of the mixture which enter the chamber

$$\Delta I = uI_o = \frac{uA\rho R}{m}\,. \tag{11}$$

The expressions for the mass flow rate can be obtained from (10, 11) when multiplying by a factor kT.

To illustrate the change in the particle flow through the chamber caused by etching, ΔI has been calculated for the etching rate 1 μm/min. A maskless aluminium layer on a 3 inch Si wafer was considered. If $u = -1$ then $\Delta I = -4.67 \times 10^{18}\ \mathrm{s^{-1}}$, which corresponds to a volumetric flow rate decrease by an amount of 11.6 sccm which is a value comparable with the gas flow rate through the etching chamber.

Experimental

For this study a single wafer parallel plate plasma reactor was used. Two sets of independent nozzles provided a uniform distribution of the etching gas and of pumping of the etching products. The flow and the composition of the mixture $\mathrm{SiCl_4} + \mathrm{Cl_2}$ were controlled by mass flow controllers. The gas pressure was measured by the capacitance vacuummeter MKS Instruments Baratron 221A. The reactor was pumped by a rotary pump equipped with a liquid nitrogen trap. The bottom electrode was driven by an RF generator and the upper electrode was grounded. Aluminium layer of thickness 1 μm was evaporated on Si wafer (3 inch in diameter). The wafer was positioned on the bottom electrode.

Fig. 1 shows the variation of the $\mathrm{SiCl_4} + \mathrm{Cl_2}$ total pressure (flows 10.2 and 10.6 sccm) during aluminium layer etching. The initial value of the pressure p_o before the etching is given by the gas flow rate and the pumping speed. After ignition of the RF discharge (13.56 MHz, 70 W) and the consequent removal of the

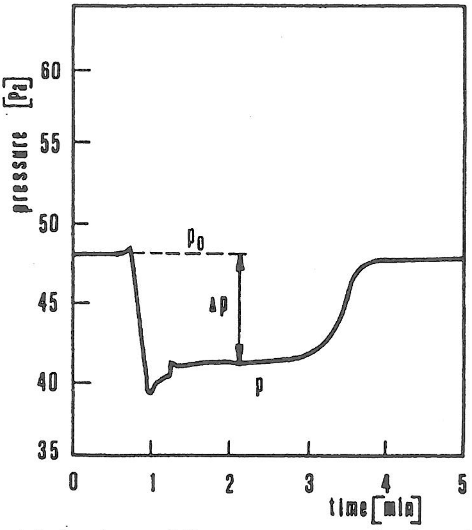

Fig. 1: Typical behaviour of the gas pressure during etching of Al layer.

native oxide layer, the chemical etching of Al layer is initiated. After 25 seconds the discharge is switched off and chemical etching continues, while the pressure decreases to a value p. The pressure remains constant until the substrate surface is cleaned of aluminium. Then the pressure starts to rise back to the initial value p_o again.

Results and discussion

The pressure difference Δp was measured in the mixture $SiCl_4 + Cl_2$ at the constant $SiCl_4$ flow (10.4 sccm) and for various Cl_2 flow rates ($3.7 \div 35$ sccm) corresponding to the total gas pressure interval $46 \div 104$ Pa. As a result, the etching rate varied from 0.2 to 1.4 μm/min.

The dependence of the etching rate on Cl_2 gas flow rate is shown in Fig. 2. As we can see from this figure, the etching rate is proportional to the flow of chlorine into the reactor. The linear behaviour indicates that the etching is limited by Cl_2 transport. It is evident that the wafer temperature (45°C) is sufficient enough to remove the etching products from Al surface. Thus, the coverage of Al surface by the etching products is negligible also at high etching rate ($\sim 1\mu$m/min). In this case Cl_2 molecules are effectively trapped by Al surface and a strong loading effect is observed in whole range of Cl_2 flow rate.

Fig. 3 shows that the gas pressure difference increases with increasing etching rate. The measured values of Δp are in the range $3 \div 28$ Pa. According to the above described theory, the gas flow rate difference should be proportional to the etching rate. Nonlinear character of the dependence in Fig. 3 is caused by nonlinear

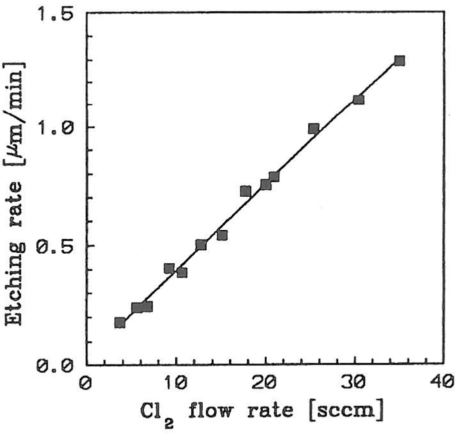

Fig. 2 Aluminium etching rate as a function of chlorine flow rate.

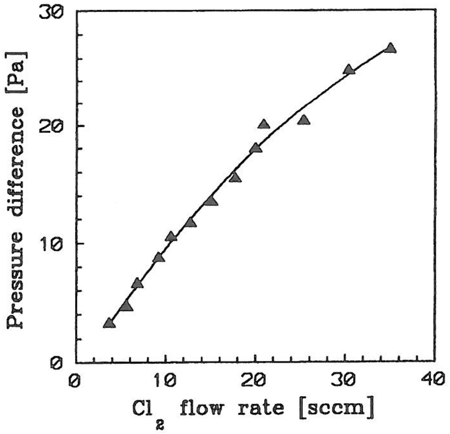

Fig. 3 Dependence of gas pressure difference on chlorine flow rate.

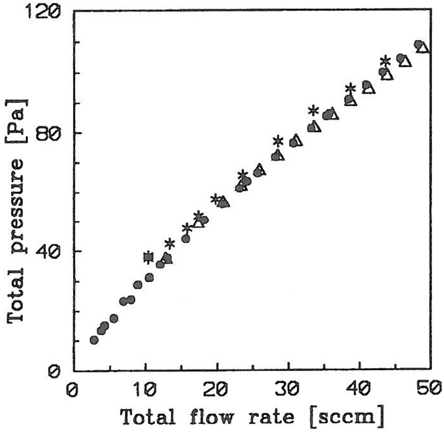

Fig. 4 Gas flow characteristics for various mixtures: Cl_2 (●), $SiCl_4+Cl_2$ (Δ – for Q_{Cl_2}=const., * – for Q_{SiCl_4}=const.).

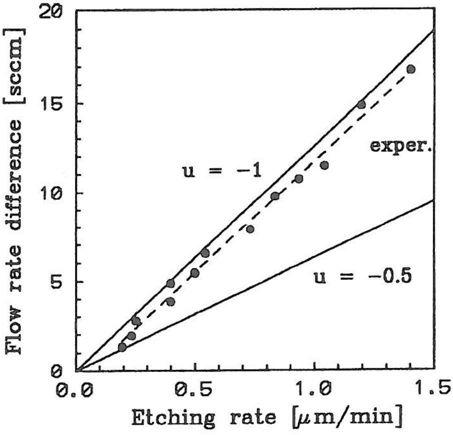

Fig. 5 Flow rate difference vs Al etching rate (points: experiment, solid lines: theory for u=−1 and u=−0.5).

dependence of the total gas pressure on the gas flow rate at the viscous flow.

The $p - Q$ dependences measured for Cl_2 and for mixtures $SiCl_4$ and Cl_2 are shown in Fig. 4. All curves are nonlinear owing to the decrease of the vacuum resistance with the flow rate at the viscous flow and they differ a little each other. We have calculated an effective viscous coefficient for Cl_2 and used mixtures. The performed analysis showed that higher viscous coefficient for Cl_2 in comparison with $SiCl_4$ is compensated by higher molecular conductance of the orifice in the angle valve.

Due to the loading effect during Al etching the concentration of Cl_2 molecules is negligible. There is only a mixture of $SiCl_4$ and Al_2Cl_6 at the reactor outlet. The calculated decrease of the viscous coefficient is compensated by the effect of lower molecular conductance for Al_2Cl_6 molecules. Therefore we have assumed that the flow characteristic of mixture $SiCl_4 + Al_2Cl_6$ is also near to those as shown in Fig. 4. This conclusion was checked by the $p - Q$ characteristic measured for the etching mixture.

Using the flow characteristics for $SiCl_4 + Cl_2$ shown in Fig. 4, the pressure dependence was transformed into equivalent gas flow differences at the outlet of etching chamber. The obtained values ΔQ are plotted in Fig. 5 as a function of the etching rate. This dependence is linear and yields a value of u equal to -1. Thus, the evidence of the reaction (8) was obtained for chemical etching of Al in $SiCl_4 + Cl_2$ at 45°C. This agrees well with the conclusion of theoretical calculations and experimental results of other authors [2, 3].

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