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Analysis of Particle Contamination in Plasma Reactor by 2-Sized Particle Growth Model

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Abstract−Rapid particle growth in the silane plasma reactor by coagulation between 2-sized particles was analyzed for various process conditions. The particle coagulation rate was calculated considering the effects of particle charge distribution based on the Gaussian distribution function. The large size particles are charged more negatively than the small size particles. Some fractions of small size particles are in neutral state or charged positively, depending on the plasma conditions. The small size particle concentration increases at first and decreases later and reaches the steady state by the balance of generation rate and coagulation rate. The large size particles grow with discharge time by coagulation with small size particles and their size reaches the steady state, while the large size particle concentration increases with discharge time by faster generation rate and reaches the steady state by the balance of generation and disappearance rates. As the diameter of small size particles decreases, the diameter of large size particles increases more quickly by the faster coagulation with small size particles of higher concentration. As the residence time increases, the concentration and size of large size particles increase more quickly and the average charges per small size and large size particle decrease.

Key words: 2-Sized Particle Growth Model, Plasma Contamination, Small Size Particles, Large Size Particles, Gaussian Charge Distribution Function

INTRODUCTION

Plasma discharge processes are quite notorious from the point of particle contamination and those particles can induce several serious effects on the performance and quality of microelectronic devices and also on the final product cost. Particles in size from few nanometers to microns are usually found inside the plasma reactor. Some contaminating particles originate outside the plasma process and other particles can be formed inside the plasma. There are two sources of particles formed inside the plasma: one is homogeneous formation in the gas phase due to the plasma chemistry, and the other is heterogeneous formation due to the fracture of deposited thin films. Those particles are believed to grow by coagulation and condensation. The particles in the plasma reactor are usually charged negatively and most of those particles are found at the plasma sheath boundaries where several forces on the particles are balanced. The particle contamination problems in plasma reactor have been widely analyzed on the experimental/theoretical basis because of their enormous economic impacts on semiconductor industries [Bouchoule and Boufendi, 1994; Boufendi and Bouchoule, 1994; Childs and Gallagher, 2000; Howling et al., 1993; Huang and Kushner, 1997; Kortshagen and Bhandarkar, 1999; Selwyn, 1993, 1994; Shiratani et al., 1994, 1996; Watanabe, 1997].

Howling et al. [1993] measured the particle sizes and concentrations in silane and Ar plasmas by the LLS and also modeled the agglomeration phase by the Brownian free molecular coagulation model. Watanabe et al. [Shiratani et al., 1994, 1996; Watanabe, 1997] analyzed the particle growth in plasma reactor by the Laser Light Scattering (LLS) methods and proposed that the particles in the plasma reactor follow three phases (initial growth phase, rapid growth phase, growth saturation phase) to grow to submicron sizes. Bouchoule et al. [Bouchoule and Boufendi, 1994; Boufendi and Bouchoule, 1994] suggested the particle growth kinetics for particle sizes from 2 nm to a few 100 nm in a rf-argon-silane plasma and reported that the particles grow rapidly by the coagulation in the first phase and slowly by the surface deposition process on independent particles in the second phase. Horanyi and Goertz [1990] considered theoretically the particle growth by enhanced coagulation between the oppositely charged, differently sized grains in plasma region and suggested that if the ionization fraction is $\ll 10^{-13}$, the enhanced coagulation might be the most important process responsible for grain growth in the size range of 0.1-500 μm. Kortshagen and Bhandarkar [1999] studied the growth of nanometer particles in low pressure plasmas and showed that particle coagulation is enhanced compared to coagulation in neutral aerosols due to the attraction of oppositely charged particles. Childs and Gallagher [2000] studied the particle growth in pure silane rf discharge, using the LLS method, and showed that the particle density is a sensitive function of gas pressure and rf voltage. Kim and Ikewawa [1996] and Kim and Kim [1997, 2000] analyzed the particle formation, growth and transport in silane plasma reactor with the plasma chemical reactions which are important for the particle formation in silane plasma reactor and predicted the distributions of those particles inside the plasma reactor for several process conditions based on the neutral particles. Recently, Kim and Kim [2002a, b] calculated the change of particle size distribution in silane plasma reactor by applying the discrete-sectional method. They also included the particle charge distribution for each particle size, based on the Gaussian distribution function.

In this study, we assumed that the particles in the plasma reactor are composed of 2-sized (large size and small size) particles and analyzed the rapid particle growth of large size particles by coagu-

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lation with small size particles in silane plasma reactor, considering the Gaussian distribution function for particle charges. The effects of particle charge distribution on coagulation were considered to calculate the particle growth by coagulation in the plasma reactor. The effects of the process conditions (small size particle diameters and residence times) on the rapid particle growth were investigated.

THEORY

The particles in plasmas are found to be divided into 2-sized groups, small and large size particles [Bouchoule and Boufendi, 1994; Boufendi and Bouchoule, 1994; Kim and Kim, 1997, 2000; Kim and Ikegawa, 1996; Shiratani et al., 1994, 1996; Watanabe, 1997]. Fig. 1 shows the particle growth model of 2-sized particles in this study to predict the rapid growth of large size particles in plasma reactor. All the small and large size particles in plasma reactor will be charged or in neutral state. The average electron charge on particle is proportional to particle diameter and the large size particles will be charged more negatively than the small size particles. The smaller particles (d≤10's nm) can have more possibility of being neutral or even being charged positively, depending on the plasma conditions [Kim and Kim, 2002a, b]. The particles of opposite charges will collide with each other very fast and the neutral particles can collide with all particles, but particles of same charges cannot collide together (Fig. 1). We calculated the rapid growth of large size particles by coagulation with small size particles in the plasma reactor, taking into account the particle charge distributions.

Most of those particles in plasmas are charged negatively to balance the currents onto the particles by slowly moving ions and fast moving electrons and the particle charging affects the particle growth by coagulation in plasma reactor significantly. Based on the analysis by Matsoukas et al. [1996], upon collision with positive and negative gas-phase charges, the particle undergoes a stepwise change in either direction. Schematically, the charging process of particles in plasma reactor is described by the sequence of steps shown below:

$$f(q-1) \underset{I_e(q)}{\overset{I_+(q-1)}{\rightleftarrows}} f(q) \underset{I_e(q+1)}{\overset{I_+(q)}{\rightleftarrows}} f(q+1), \quad (1)$$

$$\frac{\partial f(q)}{\partial t} = I_+(q-1)f(q-1) + I_e(q+1)f(q+1) - [I_e(q) + I_+(q)]f(q). \quad (2)$$

Matsoukas et al. [1996] solved the population balance equation for particle charging [Eq. (2)] and gave us the Gaussian distribution function [Eq. (3)] for particle charges. The average charge ($\bar{q}$) and variance (σ^2) of the distribution were expressed in terms of plasma parameters by Eqs. (4) and (5), respectively [Matsoukas et al., 1996; Kim and Kim, 2002a, b]:

$$f(q) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{(q-\bar{q})^2}{2\sigma^2}\right], \quad (3)$$

$$\bar{q} \approx C\frac{2\pi\varepsilon_0 d k_B T}{e^2} \ln\frac{N_+}{N_e}\left(\frac{M_e T_e}{M_+ T_+}\right), \quad (4)$$

$$\sigma^2 = \left(\frac{1}{\beta_e}\right)\left(\frac{1-t'\beta_e\bar{q}}{t'+1-t'\beta_e\bar{q}}\right), \quad (5)$$

where $\beta_e = \frac{e^2}{2\pi\varepsilon_0 dkT}$ and $t' = \frac{T_e}{T_+}$.

In Eq. (4), the particle average charge ($\bar{q}$) is given as a function of particle diameter (d), number concentrations of electron and positive ion (N_e, N_+), masses of electron and positive ion (M_e, M_+) and temperatures of electron and positive ion (T_e, T_+).

To calculate the particle growth in a plasma reactor, we neglected the coagulations between the particles of same charges and included the coagulations between the charged particles and the neutral particles and between the neutral particles and between the oppositely charged particles. Based on the Gaussian charge distribution function [Eq. (3)], we calculated the fractions of neutral, negatively charged and positively charged particles (F_{neu}, F_{neg}, F_{pos}) and also the average charges of the negatively and positively charged particles ($\bar{q}_{neg}$, $\bar{q}_{pos}$) for various plasma conditions [Kim and Kim, 2002a, b] and used to calculate the particle coagulation rates inside the plasma

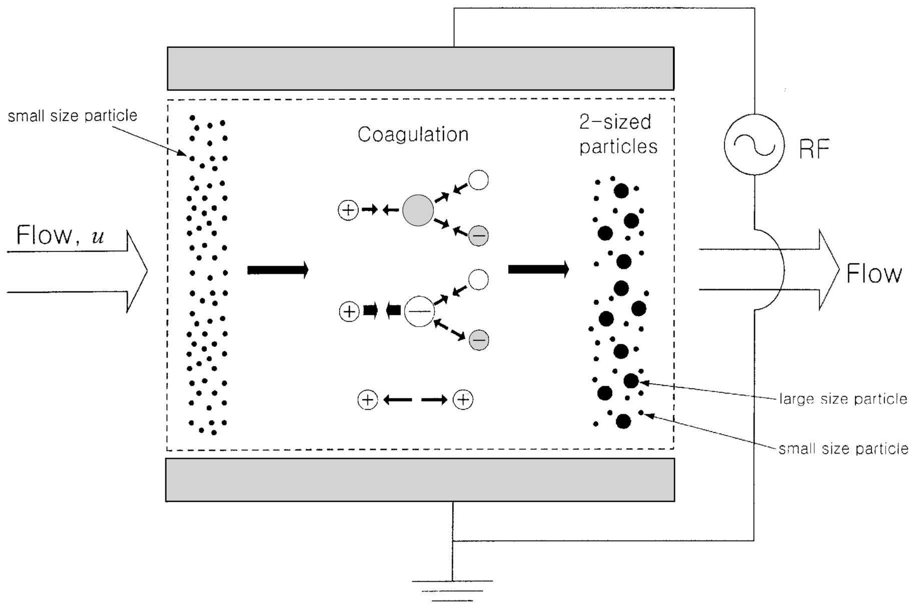

Fig. 1. Particle growth model of 2-sized particles in silane plasma reactor.

reactor. The model equations of rapid particle growth by coagulation are based on the assumption of monodisperse distributions of large and small size particles, respectively, as observed by several experiments [Bouchoule and Boufendi, 1994; Boufendi and Bouchoule, 1994; Shiratani et al., 1994, 1996; Watanabe, 1997]. The plasma reactor is assumed to be a continuously stirred tank reactor. The governing equations for large size particle volume (θ), large size particle concentration (n_p) and small size particle mass concentration (ρ_0) can be expressed by Eqs. (6)-(8), respectively.

$$\frac{d\theta}{dt}=\frac{\rho'_p}{\rho_d}-\frac{\theta}{\tau}+\frac{E_{coag}}{\rho_d}, \tag{6}$$

$$\frac{dn_p}{dt}=\frac{\rho'_p}{\rho_d v_{p,0}}-\frac{n_p}{\tau}, \tag{7}$$

$$\frac{d\rho_0}{dt}=\rho'_0-\frac{\rho_0}{\tau}-E_{coag}, \tag{8}$$

$$E_{coag}=[F_{p,neu}F_{0,neu}+F_{p,neu}F_{0,neg}+F_{p,neu}F_{0,pos}+F_{p,neg}F_{0,neu}+F_{p,neg}F_{0,pos}(1-\Gamma)+F_{p,pos}F_{0,neu}+F_{p,pos}F_{0,neg}(1-\Gamma)]\beta\rho_0(t)n_p, \tag{9}$$

$$\Gamma=\frac{\overline{Q}_{p,neg\ or\ pos}\overline{q}_{0,pos\ or\ neg}e^2}{\pi\varepsilon_0 m_R v_R^2(D_p+d_0)}. \tag{10}$$

The first term on right hand side (RHS) of Eq. (6) shows the effect of large size particle generation rate, and the second and the third terms, the effect of the disappearance rate by particle coagulation and by fluid convection, respectively. Eq. (7) shows the effects of large size particle generation and fluid convection on the change of large size particle concentration (n_p). The RHS of Eq. (8) shows the change of small size particle mass density by the small size particle generation, the small size particle disappearance by coagulation with large size particles, and by fluid convection, respectively. $(1-\Gamma)$ in Eq. (10) is the enhancement factor of particle coagulation induced by the Coulomb force between the oppositely charged particles [Lieberman and Lichtenberg, 1994].

We also included the electroneutrality condition in plasma reactor by Eq. (11), considering the charges by positive ions, negative ions, large size particles and small size particles.

$$N_+=N_-+N_e-n_p\overline{Q}_p-n_0\overline{q}_0. \tag{11}$$

We assumed the positive and negative ion concentrations in plasma reactor are constant and calculated the electron concentration by the Newton-Raphson method [Riggs, 1988]. The electroneutrality condition [Eq. (11)] might not be satisfied in the sheath region of the plasma reactor, but can be satisfied in the bulk plasma and approximately in the sheath boundary region where most of those particles are usually found.

The governing equations of Eqs. (6)-(8) were solved numerically by the ODE solver, DGEAR subroutine, to calculate the large size particle volume and the large size and small size particle concentrations in plasma reactor. In every time step of integration, the electroneutrality condition [Eq. (11)] was also solved to calculate the electron concentration (N_e). The particle charge distributions [Eq. (3)], the fractions of negatively charged, neutral or positively charged particles and the average charges of large and small size particles were calculated from the electron concentration and the coagulation rates between large and small size particles were calculated.

RESULTS AND DICUSSIONS

The plasma conditions for ion temperature (T_+) and electron temperature (T_e) were assumed to be 300 K and 2 eV, respectively. The mass generation rates of small and large size particles were 3.0×10^{-7} g/cm^{3}s and 6.42×10^{-9} g/cm^{3}s, respectively. The concentrations of positive ions (N_+) and negative ions (N_-) were found by the numerical program [Kim and Kim, 1997] to calculate the concentration distributions of chemical species in silane PCVD for the conditions of P=0.6 Torr, T_+=300 K and Q=30 sccm and were about 6.0×10^{10} cm^{-3} and 5.0×10^{9} cm^{-3}, respectively. The changes of particle size and particle concentration, particle charge distribution in the plasma reactor were investigated, changing several process conditions such as small size particle diameter (d_0) and residence time (τ). The standard conditions for these variables were 10 nm, 0.617 s (30 sccm), respectively, which were the experimental observations by Shiratani et al. [1996]. The sizes of small size particles were observed to change in nms depending on the process conditions in the plasma reactor [Bouchoule and Boufendi, 1994; Boufendi and Bouchoule, 1994; Shiratani et al., 1994, 1996; Watanabe, 1997] and were changed in the range of 1-10 nm in this study. The residence times of gas stream in plasma reactor were changed in the range of 0.1854 (100 sccm)-1.854 (10 sccm) s.

Fig. 2 shows the changes in concentrations of small size and large size particles for various diameters of small size particles with time for the constant mass generation rate of small size particles ($=3.0\times10^{-7}$ g/cm^{3}s). The concentration of small size particles increases in the beginning by the faster generation rate than the disappearance rate by coagulation, reaches a maximum and decreases later by a

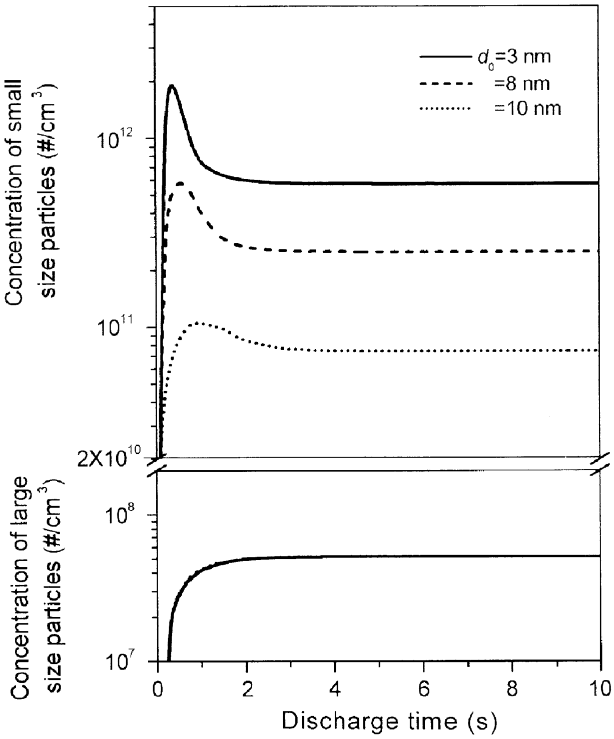

Fig. 2. Concentrations of small and large size particles for various diameters of small size particles as a function of discharge time (τ=0.617 s).

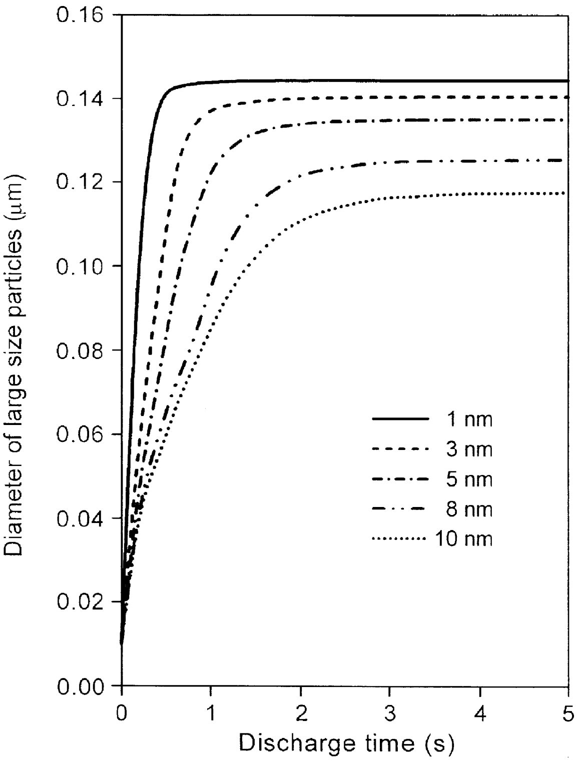

Fig. 3. Diameters of large size particles for various diameters of small size particles as a function of discharge time (τ= 0.617 s).

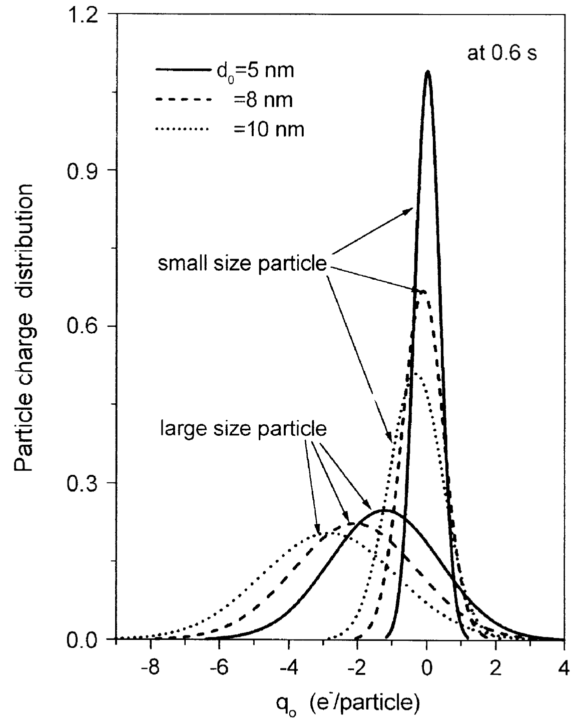

Fig. 4. Particle charge distributions of small and large size particles in plasma reactor for various diameters of small size particles (τ=0.617 s).

faster coagulation rate with large size particles. In the end of the particle growth (t≥3 s), the concentration of small size particles reaches the steady state by the balance between the generation rate and the disappearance rate by fluid convection and coagulation with large size particles. The concentration of large size particles increases in the beginning of the particle growth because the generation rate of large size particles is greater than the disappearance rate by fluid convection and reaches the steady state by the balance between the generation and the disappearance rates. As the diameter of small size particles decreases for the constant mass generation rate of small size particles, more small size particles are generated and the concentration of small size particles increases, but the large size particle concentration does not change significantly. Fig. 3 shows the changes in diameter of large size particles for various diameters of small size particle as a function of time. In the beginning, the diameter of large size particles increases quickly by the fast coagulation with small size particles. Later, as concentration of the small size particles decreases, the diameter of large size particles increases slowly and reaches the steady state. The smaller the diameter of small size particles is, the higher the concentration of small size particles is, and the diameter of large size particles becomes larger and reaches steady state more quickly.

Fig. 4 shows the charge distributions of small and large size particles for various diameters of small size particles. The large size particles are more negatively charged than the small size particles, as proposed by the Gaussian distribution function [Eq. (3)]. The total charges on large size particles ($\overline{Q}_p n_p$) and small size particles ($\overline{q}_0 n_0$) for the small size particle diameter of 10 nm are about −1.15 $\times 10^8$ e and -5.24×10^{10} e at 0.6 s, respectively. The electron concentration at 0.6 s was 2.47×10^9 and we can see that most of negative charges are located onto the small size particles. By Eq. (4), the average charges on particles in the plasma reactor increase with particle size. Because the diameter of large size particles increases with time, the large size particles become more negatively charged. As the diameter of small size particles decreases, the diameter of large size particles increases (Fig. 3) and the electron concentration to satisfy the electroneutrality condition decreases. For these conditions, the average charges on large size particles are more sensitive to the change in electron concentration than to the change in diameter of large size particles. As a result, the average charge per large size particles increases as the particle size increases. As the diameter of small size particles decreases, the average charge per large size particle decreases. Based on the Gaussian distribution function of particle charges, some fractions of small size particles can be in neutral state or can be charged positively, depending on the process conditions in the plasma reactor. The small size particles in neutral state or charged positively can collide with the large size particles charged negatively and they play an important role for the rapid growth of large size particles in the plasma reactor. As the diameter of small size particles decreases, total charges on large size particles and small size particles increase, and the electron concentration decreases and the average charges per small size particles and large size particles decrease.

The changes in diameter of large size particles are shown for various residence times in Fig. 5. To analyze the effects of residence time on the properties of large size and small size particles in plasma reactor, we changed the total gas flow rate from 10 sccm (τ= 1.854 s) to 100 sccm (τ=0.1854 s). The diameter of large size particles increases with discharge time by coagulation with small size particles. As the residence time of the gas stream increases, the large size particles can grow for longer time inside the plasma reactor and the size of large size particles becomes larger. The changes in concentrations of small and large size particles are shown for various residence times in Fig. 6. The concentration of large size particles increases in the beginning of the particle growth because the generation rate of large size particles is greater than the disappearance rate by fluid convection and reaches the steady state later by the balance between the generation and the disappearance rates. With the increase of residence time, the large size particles can stay longer inside the plasma reactor and the concentration of large size particles increases. As the residence time decreases, the disappearance rate of small size particles by fluid convection increases and the concentration of small size particles increases more slowly in the beginning of the particle growth. With the increase of residence time, the coagulation rate between large size and small size particles becomes faster because of the larger particle size and higher concentration of large size particles (Figs. 5 and 6), and the concentration of small size particles becomes lower at steady state, which was also shown by the experiments by Shiratani et al. [1996]. For the residence times of 1.854 s and 0.617 s, the concentration of small size particles increases in the beginning of the particle growth because the generation rates of small size particles are greater than the disappearance rates by coagulation with large size particles and by fluid convection. The concentration of small size particles reaches the maximum and decreases later by the faster coagulation rates with large size particles and reaches steady state. For the residence times of 0.232 s and 0.185 s, the effect of fluid convection becomes more significant than the effect of particle coagulation and the concentration of small size particles increases with discharge time and reaches steady state without showing the maximum peak. Fig. 7

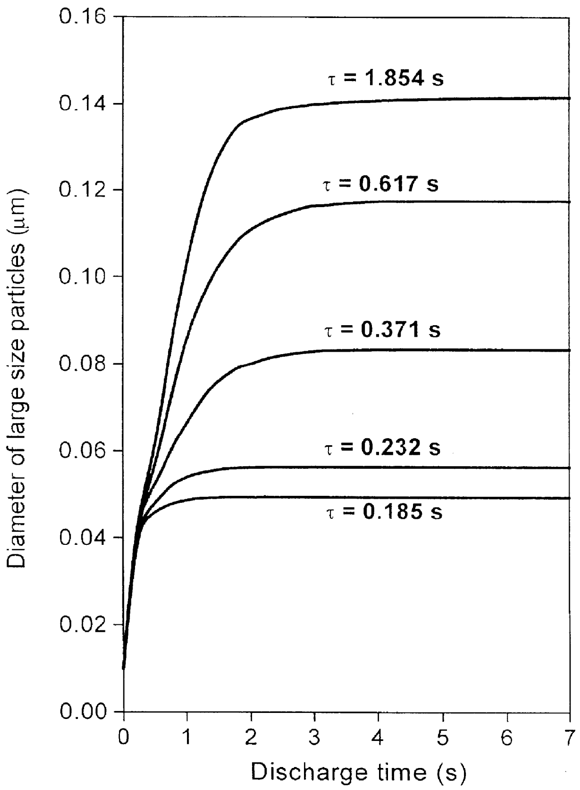

Fig. 5. Diameters of large size particles for various residence times as a function of discharge time (d_0=10 nm).

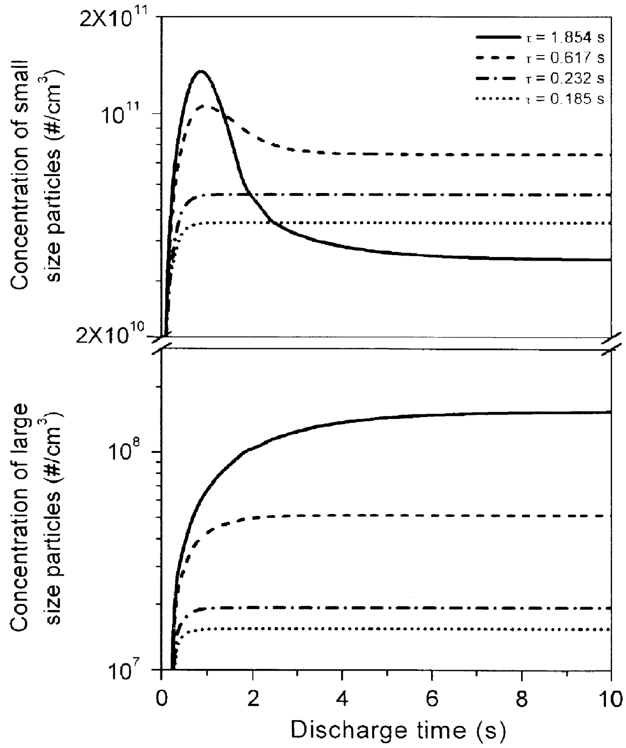

Fig. 6. Concentrations of small and large size particles for various residence times as a function of discharge time (d_0=10 nm).

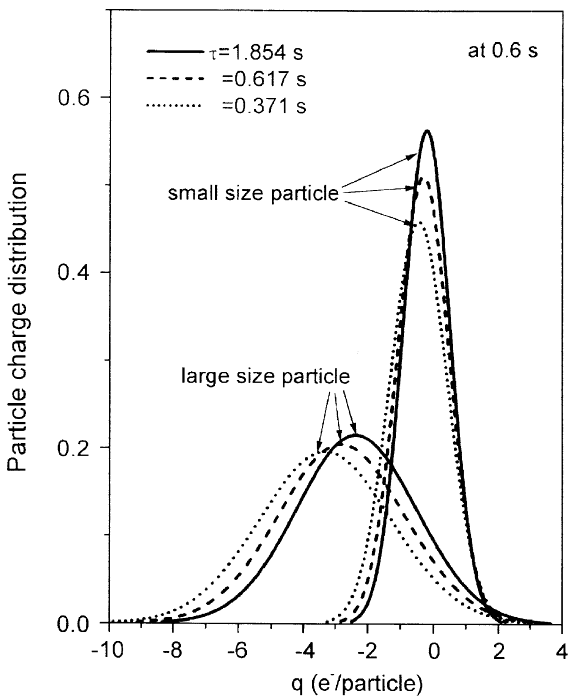

Fig. 7. Particle charge distributions of small and large size particles in plasma reactor for various residence times (d_0= 10 nm).

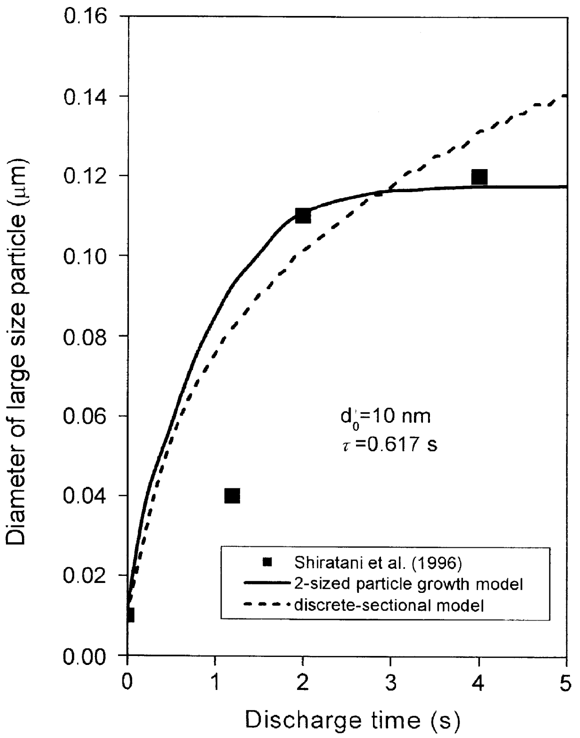

Fig. 8. Comparison of predicted large size particle diameters by 2-sized particle growth model and by discrete-sectional model [Kim and Kim, 2002a] with experimental results by Shiratani et al. [1996] (τ=0.617 s).

shows the particle charge distributions of small size and large size particles at 0.6 s for various residence times. Again, the large size particles are charged more negatively than the small size particles. As the residence time increases, total amount of particles and total charges on particles increase and, as a result, the electron concentration decreases and the average charges per small and large size particles decrease.

In Fig. 8, the model results of the diameter of large size particles in this study (particle growth model by 2-sized particles) were compared with the model results of the discrete-sectional model [Kim and Kim, 2002a, b] and the published experimental results by Shiratani et al. [1996] for standard conditions. In the model results of 2-sized particles and the experimental results, the diameter of large size particles increases with discharge time by coagulation with small size particles and reaches steady state, but the diameter of large size particles in the discrete-sectional model grow continuously because more of the large size particles are remaining inside the plasma reactor. The results for the diameters of large size particles by the 2-sized particle growth model here were found to be in close agreement with the experimental data by Shiratani et al. [1996].

CONCLUSIONS

We analyzed the rapid particle growth in the silane plasma reactor by 2-sized particle growth model, assuming that the particles are composed of large and small size particles. The particle charge distribution was calculated by the Gaussian distribution function. We investigated the effects of process conditions during rapid particle growth.

It is usually believed that most of the particles in a plasma reactor are charged negatively, but we found that some fractions of particles can be in the neutral state or can be charged positively, depending on the process conditions in plasma reactor. The large size particles are charged more negatively than the small size particles. Some fractions of small size particles are in the neutral state or charged positively depending on the plasma conditions, and the small size particles charged positively can collide very fast with the large size particles charged negatively.

The small size particle concentration increases at first and decreases later and reaches the steady state by the balance of generation rate and coagulation rate. The diameter and concentration of large size particles increase with discharge time and reach the steady state. As the small size particle concentration increases, the amount of negative charges on the small size particles increases, and the electron concentration in the plasma decreases to satisfy the electroneutrality condition and the particles in plasmas are charged less negatively. The small size particle concentration increases with the decrease of small size particle diameter, and the diameter of large size particles increases more quickly by faster coagulation with small size particles. The concentration and diameter of large size particles increase with the increase of residence time. The total amount of particles increases with the increase of residence time and the electron concentration in plasmas decreases because more electrons are absorbed onto the particles and the particles are charged less negatively. The model results in this study were in good agreement with the published experimental data [Shiratani et al., 1996].

ACKNOWLEDGMENTS

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NOMENCLATURE

C : constant, 0.73
d_0 : diameters of small size particles [cm]
D_p : diameters of large size particles [cm]
e : elementary charge of electron [C]
f(q) : particle charge distribution function
F_{neu}, F_{neg}, F_{pos}: fractions of neutral, negatively charged and positively charged particles
E_{coag} : increase rate of large size particle mass by coagulation with small size particles
I(q) : flux of species which pass through the q particle charges
k_B : Boltzmann constant, 1.38×10^{-16} [gcm^2/sec^{2}K]
m_R : reduced mass between the moving particles
M : mass of chemical species [g]
n : particle number concentration [cm^{-3}]
N : number concentrations of chemical species [cm^{-3}]
q : particle charges [e]
$\bar{q}_{neg}$, $\bar{q}_{pos}$: average charges of the negatively and positively charged particles [e]
$\bar{q}_0$: average charges of small size particles [e]
$\bar{Q}_p$: average charges of large size particles [e]
t : time [s]

T_+ : temperature of positive ions [K]
T_e : electron temperature [eV]
$v_{p,0}$: volume of a small size particle
v_R : relative velocity between the moving particles

Greek Letters

β : collision frequency function between particles [Friedlander, 1977]
ε_0 : permittivity of free space, 8.854×10^{-21} [C^2/dyncm^2]
θ : dimensionless totoal volume of large size particles
ρ_d : particle density [g/cm^3]
ρ'_p : mass generation rates of large size particles
ρ_0 : mass concentration of small size particles [g/cm^{-3}]
ρ'_0 : mass generation rates of small size particles
τ_{res} : residence time [s]

Subscripts

0 : small size particle
e : electron
p : large size particle
+ : positive ion
– : negative ion

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