

## SURFACE CLEANING BY ICE-PARTICLE JET(II) - PREPARATION OF CONTAMINATED SURFACE AND ITS CLEANING -

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**Abstract** – A physical method of surface cleaning has been developed using the jet of micron-sized ice particles in a low pressure impactor. The substrate to be cleaned was prepared by both applying organic films and depositing polystyrene (PS) particles on substrates. The PS particles, as a source of contamination, were produced with the average sizes ranging from 0.5 to 3 micrometers and narrow size distribution under well-controlled conditions. The particles were deposited uniformly and controllably by changing the deposition time, carrier gas flow rate, and substrate temperature. Major variables for removing both the films and contaminating particles were the removal time, chamber pressure, carrier gas flow rate, nozzle-to-substrate distance and number concentration of ice particles. We proposed two models on film-removal mechanism, which could be applied, depending on the kinetic energy of the ice particles and the hardness of the film. The ice-particle jet system, as a method of cleaning, was found so effective to remove physically both films and particles adhered to a wafer.

**Key words:** *Ice-Particle Jet, Wafer Cleaning, Film Removal, Monodisperse Particles, Low-Pressure Impactor, Particle Deposition*

### INTRODUCTION

In recent LSI (Large Scale Integration) and LCD (Liquid Crystal Display) manufacturing processes, the demand of effective cleaning method has been increased, for contaminants such as resist residue, submicron particles, organic films and native oxide molecules are major factors that reduce the yield and degrade the device quality. Several methods are presently used to clean surfaces for the electronics industry [Skidmore, 1987; Ruzyllo, 1990]. Solvent or chemical cleaning is used to remove contaminant films from surfaces. Chemical solutions can be combined with megasonic or ultrasonic cleaners to enhance cleaning efficiency. However, solvent or chemical cleaning requires extremely pure and clean agents. In addition, the agent becomes progressively more contaminated as it is used and must be disposed of periodically. Disposal of such agents frequently causes environmental problems. Also, such agents require special safety treatments in order to minimize exposure to operators. On the other hand, gas cleaning and liquid spray cleaning are presently used to clean relatively large particles from silicon wafers. Gas jets [Otani et al., 1995] are, however, ineffective in removing particles smaller than 5 micrometers. This is because the adhesive force tending to hold the particle to the surface is proportional to the particle diameter while the drag force by the gas tending to remove the particle is proportional to the diameter squared [Hinds, 1982]. Therefore, the ratio of these forces tends to favor adhesion as the particle size reduces. Also, smaller particles are not exposed to strong drag forces in the jet since they can lie within the surface boundary layer where the gas velocity is low. Liquid jets provide stronger

shear forces to remove particles but are expensive and difficult to keep its high purity and may leave contaminating residues on drying. Also, a conventional liquid spray solvent is environmentally harmful.

Exposure to ozone combined with ultraviolet light can be used to decompose contaminating hydrocarbons from surfaces. However, this technique has not been suitable to remove contaminating particles. A recently developed cleaning technique involves the use of ice particles [Ohmori et al., 1989; Endo et al., 1992], carbon dioxide snow [Peterson, 1994], solid argon or nitrogen particles [McDermott et al., 1992] to sandblast contaminated surface area. This new cleaning method is done by jetting fine and ultra clean solidified particles onto the surface of semiconductor wafers. The method overcomes the disadvantages of the conventional techniques by providing a highly pure and inert particulate aerosol for cleaning substrates, while avoiding recontamination by the cleaning particles themselves. Yet details of the processes have been rarely known, especially the quantitative effect of process variables on removal efficiency. Also the ice-particle jet used so far consists of relatively large particles (up to a couple of hundreds of micrometers) such that they sometimes damage the substrate surface significantly.

The present work is aimed at developing an effective method of removing particles and films from sensitive microelectronics surfaces by impinging a aerosol stream of fine ice particles against such surfaces. In the previous paper [Ju et al., 1996], the ice particles with different diameters were prepared by various methods and the power of particle impaction was examined by applying the particle jet onto aluminum foil or lacquer film on glass, and tracking the morphology of the surface. The present work is the extension of the previous work, in systematically preparing the surface contaminated with particles

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and films under highly controlled environment, and quantitatively studying the efficiency of their removal by high-speed jet of the fine ice particles under different process parameters.

## EXPERIMENTAL

### 1. Preparation of Contaminated Substrates

#### 1-1. Preparation of Polystyrene Particles

Monodisperse polystyrene particles, used as contaminating particles, were produced under the optimum dispersion polymerization condition determined in an article [Okubo, 1992]. The dispersion polymerization of styrene was carried out at the temperatures around 70°C for 24 hours under a nitrogen atmosphere in a four-necked, round-bottom flask with reflux. The reaction mixture consisted of 137 g of ethanol, 40 g of water, 20 g of styrene, 0.336 g of 2,2'-azobisisobutyronitrile (AIBN), and 2.40 g of poly(acrylic acid) (PAA). The mixture in the flask was stirred with an anchor-type stirrer at 60 rpm. All the chemicals were reagent-grade and no further purification was carried out on them. By varying the amount of either ethanol or water around the value given above, the effects of the media on the average size and size distribution of the particles were investigated. The size and shape of the particles were observed with scanning electronmicroscope (SEM, JSM-35CF, JEOL Co.) and optical microscope. The number and weight average sizes with size distribution were measured with laser particle size analyzer (LPA-3000, Otsuka Electronics).

#### 1-2. Particle Deposition

Schematic diagram of particle-deposition system is shown in Fig. 1. The polystyrene particles suspended in DI water were atomized by an ultrasonic nebulizer. Particle concentration in the suspension was chosen so that particle agglomeration was negligible. The diffusion dryer was used to dry the atomized droplets to leave the polystyrene particles only suspended in the carrier gas. The dryer was 5 cm in inside diameter, 10 cm in outside diameter and 70 cm long, and was filled with silica gel in the annular part of the tube. After passing through the diffusion dryer, the polystyrene particles were then supplied to the upstream wafer surface through an isokinetic injection tube in the deposition chamber. The chamber was a cylinder with the diameter of 13 cm. We used the injection tube of 37 mm

long and set the vertical distance between the injection tube and the substrate surface to be 15 cm more or less. The temperature of the substrate placed on an aluminum plate of 5-cm diameter was controlled by flowing either heating medium (hot water) or coolant (ethylene glycol) whose temperatures were controlled accurately in an external temperature bath. Five to six pieces of 5 mm×5 mm slide glass were placed on the aluminum plate around the center. The number of the particles deposited on the area of 1 mm×1 mm was counted through the optical microscope. The effects of the deposition time, carrier gas flow rate, and the substrate temperature on the number of the particles deposited were evaluated.

#### 1-3. Film Preparation

For the experiments of film removal, a film was made of either lacquer on a slide glass or photoresist on a wafer. Lacquer was applied on the glass by spraying. The thickness of the lacquer film so obtained was  $25 \pm 5$  micrometers. Also a uniform photoresist film of 10 micrometer thick was prepared on the wafer.

### 2. Cleaning Experiment

A schematic diagram of the system used in our experimental work on cleaning is shown in Fig. 2. The geometry and dimension of the apparatus were given in our previous paper [Ju, 1996]. Although other methods for generating aerosols of different droplet sizes are possible, as described in the previous study, the present system used an aerosol generated by atomizing DI water using an ultrasonic nebulizer. The carrier gas used for entraining the aerosol droplets and the bypass gas for controlling the aerosol concentration were all nitrogen. The aerosol stream passed through a solidification chamber which was surrounded by a liquid nitrogen bath. The stream, now consisting of fine ice particles and nitrogen gas, flowed out of the chamber through a 0.5 mm nozzle which formed a sonic jet in the cleaning chamber. The cleaning chamber was maintained by a vacuum pump at a reduced pressure (0-400 mmHg). The aerosol particles (typically 2-10 micrometers, as found in the previous study) in the jet are much more massive than the gas molecules and hence are largely unaffected by collisions with the background gas molecules. In this manner, a beam of sonic particles of ice is directed onto a substrate to be cleaned. A

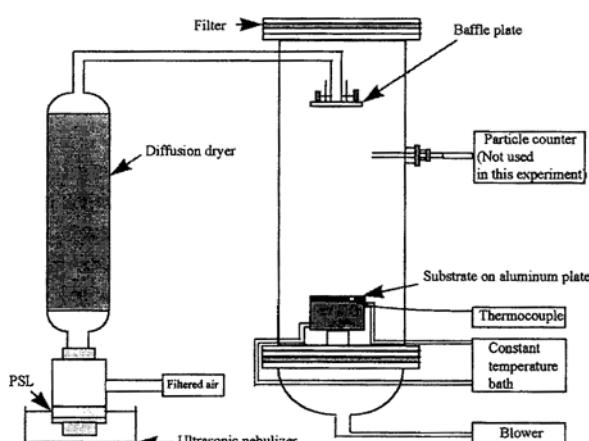


Fig. 1. Schematic diagram of particle deposition chamber.

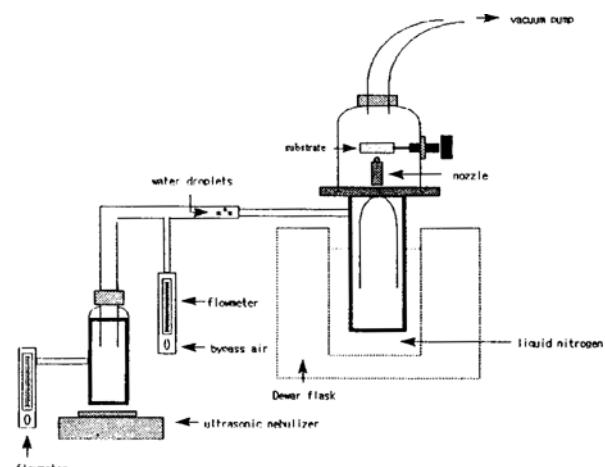


Fig. 2. Schematic diagram of cleaning chamber.

number of the process variables were studied, including the cleaning time, chamber pressure, carrier gas flow rate, nozzle-to-substrate distance and number concentration of ice particles. The particle number concentration was controlled by changing the amount of water holdup in the nebulizer. In this work, the effect of these parameters on the removal efficiency of the contaminating particles and the removal depth of the film, were investigated, respectively. The removal depth was measured with Surfscan (Tokyo Seimitsu Co.) and the % removal was evaluated by counting the numbers of particles remained on the 1 mm × 1 mm area of substrate both before and after cleaning. The surface of the substrate was observed by the SEM and optical microscope.

## RESULTS AND DISCUSSION

In order for ice-particle jet cleaning to occur, it is necessary (but not sufficient) that particles entrained in the jet have sufficient inertia to impact on the surface and not be deflected substantially by the motion of the host gas. Such an impactation process has been extensively studied, under different geometries. According to inertial impactor theory [Hinds, 1982], the efficiency, with which particles of diameter  $d_p$  and density  $\rho_p$ , in a right-angle jet impact on a surface is dependent on several parameters: the ratio of the nozzle-to-substrate distance, 1 to the nozzle inside diameter,  $d_n$ ,  $s (=1/d_n)$ ; the Reynolds number of the jet; impactor geometry; Stokes number,  $Stk: Stk=\rho_p U d_p^2 / C/18\mu d_n$ , where  $U$  is the exit jet velocity,  $\mu$  is the viscosity of the gas in the jet, and  $C$  the Cunningham slip correction factor which allows a noncontinuum effects on particle drag. An impactor is characterized by its collection efficiency as a function of particle size. The diameter of a particle which is collected with a 50 % efficiency is referred as the cutoff diameter and the corresponding Stokes number is referred as  $Stk_{50}$ . According to Marple et al. in the range of  $s$  from 1.0 to 5.0,  $Stk_{50}$  remains fairly constant. Unfortunately, there are no published data for  $s$  greater than 5.0. In our system, the smallest  $s$  used is 10, which is much larger than that employed in impactor design. In order for our cleaning system to be effective, the particles, which impact on the surface, should rebound with entraining the contaminating particles or films on the substrate. A critical velocity,  $V_c$  [Hinds, 1982] for which bounce will occur if exceeded, is defined by  $V_c=\beta/d_n$ , where  $\beta$  is a constant that depends on the materials and geometry. For example, a lower limit of velocity for which bounce will occur on uncoated metal impaction plates at  $\beta=0.02 \text{ cm}^2/\text{s}$ . Unfortunately, the  $\beta$  value for glass or wafer has not known yet. On the other hand, it was proposed [McDermott et al., 1992] that the particles must traverse the velocity boundary layer and strike the surface. A simple model assumes that the gas flow creates a boundary layer of thickness  $\delta$  having a negligible normal component of velocity. In order to strike the surface, the particles must enter the boundary layer with a normal component of velocity equal to at least  $\delta/\tau$ . The particle relaxation time  $\tau$  is given by  $\tau=d_p^2 \rho_p C / 9\mu$ . The above analysis demonstrates that the cleaning process is most effective for particles having large mass or high initial velocity. The cleaning process is also enhanced at lower pressures due to the increased particle slip and at lower gas

viscosities due to the increased decelerating drag force on the particles. Here we show the results and discuss the effect of process variables on the removal efficiency for the film and particles deposited, respectively.

### 1. Removal of Film

#### 1-1. Lacquer Film

Film removal was carried out for two types of the films-lacquer film on a slide glass and photoresist film on a wafer. The lacquer film deposited on the glass was relatively soft and could be easily removed. Fig. 3 shows the time evolution of the removal depth under the reference condition. The linear relationship shown in the figure implies that the rate of film removal is kept constant. The figure also shows the angle of impaction has negligible influence on the removal efficiency under the reference condition chosen. Fig. 4 shows the removal depth vs. chamber pressure. The lowest pressure (-400 mmHg) at which appreciable removal depth is obtained is approximately equal to the critical value at which the velocity in the throat is sonic [Smith, 1987]. Therefore, it is assumed that, in order for the ice particles to get sufficient kinetic energy for cleaning, the velocity at the nozzle should be at least sonic. The figure also shows the angle of impaction has little influence on the removal performance. The effect of nozzle-to-substrate distance on the removal depth is shown in Fig. 5. The farther the distance, the less removal depth. Because of the drag force between fluid and particles, the velocity of the ice particles relative to their host gas decreases with increasing nozzle-to-substrate distance. Therefore, for a particular chamber pressure as this distance is increased to some critical value, the particles will not have sufficient inertia to impact on the surface and cleaning will cease. Fig. 6 shows the effect of particle number concentration on the removal depth. The figure implies that as the particle number concentration increases, there appears a multiplication effect of surface softening or irritation to enhance the removal rate. The figure also shows that the right-angle and 45° impactions have no significant difference each other.

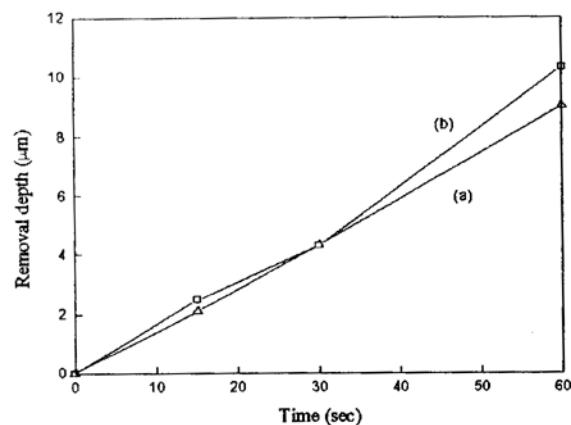


Fig. 3. Removal depth of lacquer film vs. time.

Chamber pressure=30 mmHg absolute

Nozzle-to-substrate distance=12.5 mm

Carrier gas flow rate=1,700 cc/min

Bypass gas flow rate=2,500 cc/min

Particle number concentration=23,000/cc air

Impaction angle=a: 90°, b: 45°

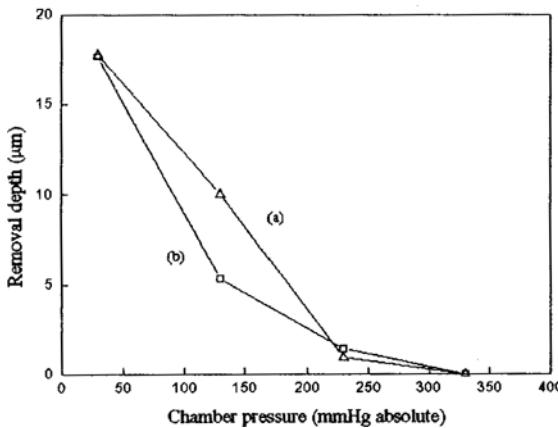


Fig. 4. Removal depth of the lacquer film vs. chamber pressure.

Impaction time=15 sec  
Nozzle-to-substrate distance=12.5 mm  
Carrier gas flow rate=1,700 cc/min  
Bypass gas flow rate=2,500 cc/min  
Particle number concentration=23,000/cc air  
Impaction angle=a: 90°, b: 45°

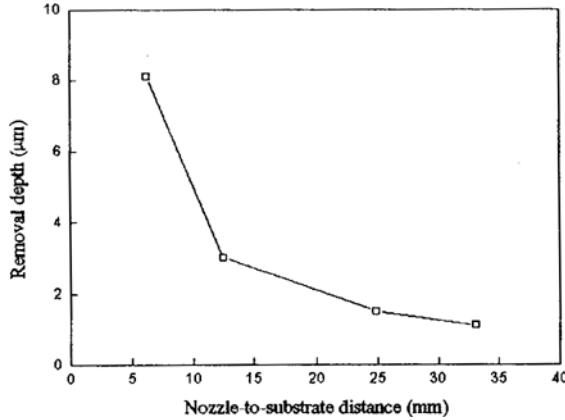


Fig. 5. Removal depth of the lacquer film vs. nozzle-to-substrate distance.

Chamber pressure=30 mmHg absolute  
Impaction time=15 sec  
Carrier gas flow rate=1,700 cc/min  
Bypass gas flow rate=2,500 cc/min  
Particle number concentration=23,000/cc air  
Impaction angle=90°

## 1-2. Photoresist Film on Wafer

In Fig. 7, the removal rate of photoresist film is also largely constant under the reference condition shown. However, in early stages the rate is somewhat lower than the average value which implies we need some time to obtain a mature surface for removal. It takes much longer to remove the same depth, compared to the case of lacquer film, since the former is much harder than the latter. The effect of other process variables on the removal depth, does not show any consistent data as described previously, when the kinetic energy of the particles is not so high. In order to reduce the cleaning time, the ice particles should be prepared with larger size, as described in the previous paper [Ju et al., 1996]. The observation, as it is, however, reveals some insight into the mechanism of removal. We

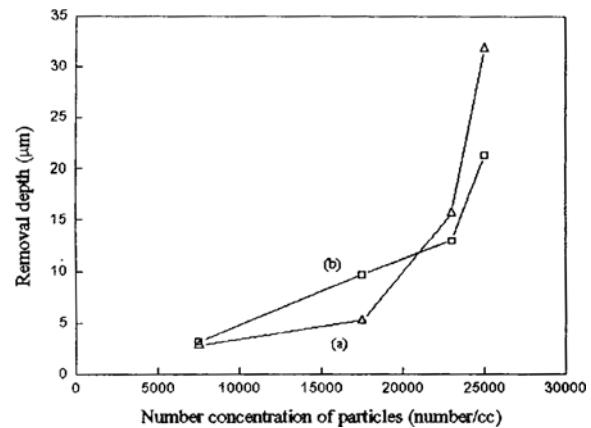


Fig. 6. Removal depth of the lacquer film vs. particle number concentration in the jet.

Chamber pressure=30 mmHg absolute  
Impaction time=15 sec  
Nozzle-to-substrate distance=12.5 mm  
Carrier gas flow rate=1,700 cc/min  
Bypass gas flow rate=2,500 cc/min  
Particle number concentration=23,000/cc air  
Impaction angle=a: 90°, b: 45°

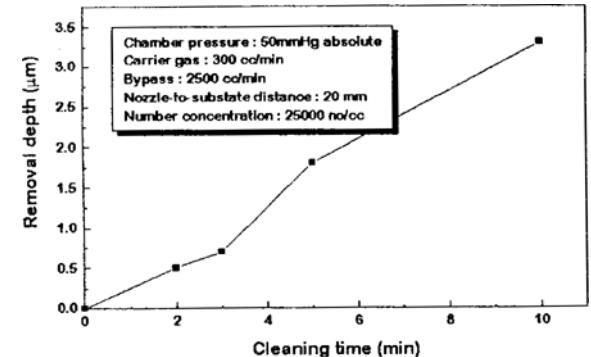
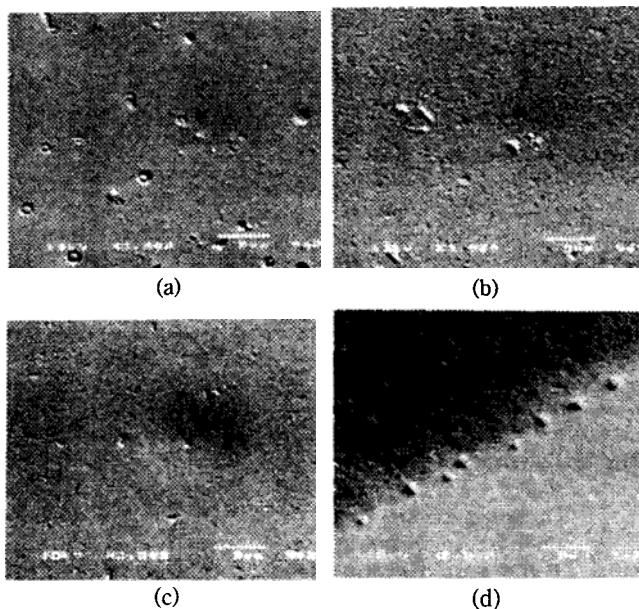


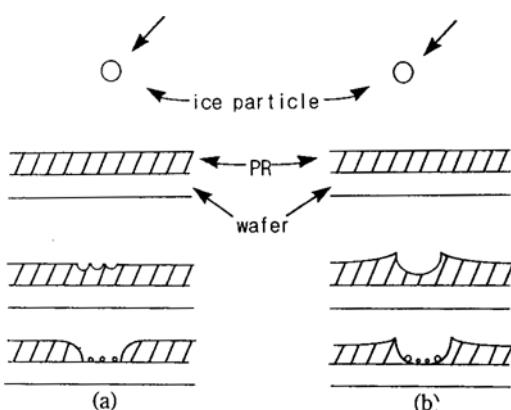
Fig. 7. Removal depth of the photoresist film vs. time.

proposed two types of removal mechanisms, depending on the hardness of the film and the kinetic energy of the impacting particles. Fig. 8(a) shows the surface of the photoresist film impacted at the distance of 5 mm from the nozzle and (b) is the surface impacted at 10 mm, both for the same cleaning time. The first type of removal mechanism prevails when the impacting particles have higher energy and the hardness of surface is not so high. The mechanism would be applied to the films shown in Fig. 8(a) as well as the lacquer film. The second type applies to the case that the kinetic energy of the impacting particles is low while the surface is comparatively harder. The SEM micrographs obeying the latter mechanism are given in Fig. 8(b), (c) and (d). In Fig. 8(d), the insufficient kinetic energy of the ice particles results in leaving, instead of removing, the residues of photoresist at the boundaries between cleaned and uncleaned regions of the wafer surface. The schematic representation of the two mechanisms is shown in Fig. 9. In model 1, the ice particles dig holes on the surface and the residues are removed from the surface. However, in model 2, the particles hits and deforms the surface, not removing it immediately. The surface is first irrigated and then broken by the



**Fig. 8. SEM micrographs of the photoresist-film surfaces during removal process.**

(a) 5 mm/2 min (b) 10 mm/2 min  
 (c) 10 mm/5 min (d) 10 mm/10 min  
 (Nozzle-to-substrate distance/removal time)



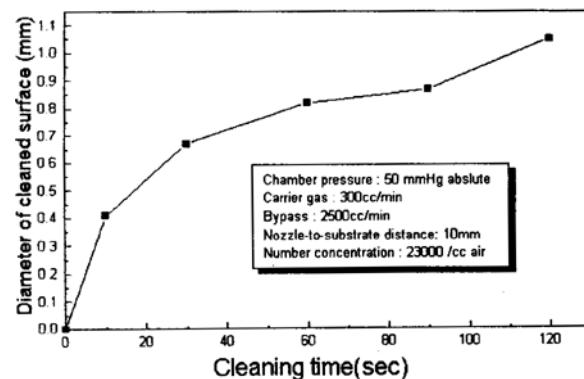
**Fig. 9. Mechanisms of ice-particle jet cleaning.**

(a) Model 1 (b) Model 2

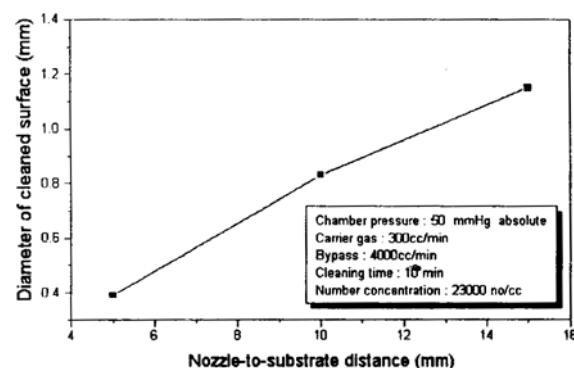
subsequent impaction of the particle jet, and finally removed. The inconsistency of the data, as described above, reflects the complicated nature of the removal mechanism of model 2. The effects of cleaning time and nozzle-to-substrate distance on the diameter of cleaning area are, however, similar to that for the lacquer film, as shown in Figs. 10 and 11. The area increases with increasing the removal time and decreasing the distance, respectively, since the number of particles, either deviating from the normal path of impaction, or impacting again after rebounding from the spot of original impaction, increases statistically.

## 2. Particle Formation and Deposition

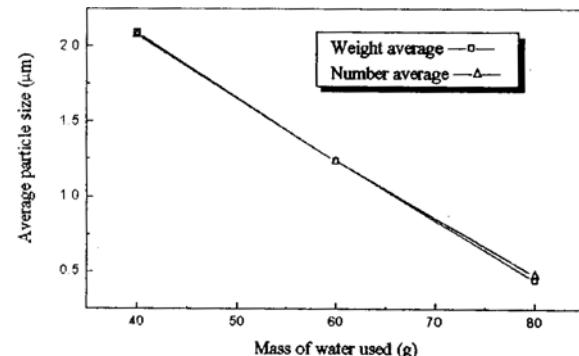
In dispersion polymerization of styrene, AIBN is used as an initiator and PAA as a stabilizing polymer. To investigate the effect of water and ethanol on the particle growth, the mass of either water or ethanol was varied around the value given un-



**Fig. 10. Diameter of cleaned surface vs. time.**



**Fig. 11. Diameter of cleaned surface vs. nozzle-to-substrate distance.**



**Fig. 12. Average size of polystyrene particles from various mass of water used.**

Reaction conditions are the same as in Table 1, except the mass of water used.

der the reference condition. Figs. 11 and 12 show the effects of the mass of water and ethanol, respectively, on both the number and weight average sizes of the polystyrene particles. The degree of coincidence of the two average sizes indicates that of monodispersity. As the mass of the media increased, both the two average particle sizes decreased together. Especially the variation of the water mass kept the monodispersity of the particles. Monodisperse particles are, in general, prepared under the controlled conditions which make the reaction rates slow and the particles grow by condensation only, suppressing excessive nucleation and coagulational growth [Ramsey, 1994]. In this respect, the role of PAA[poly(acrylic acid)], the sta-

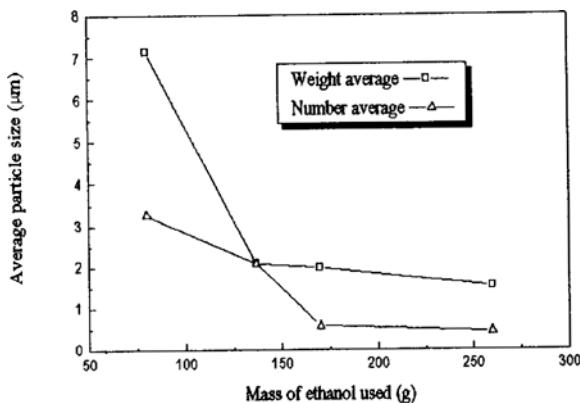


Fig. 13. Average size of polystyrene particles from various mass of water used.

Reaction conditions are the same as in Table 1, except the mass of ethanol used.

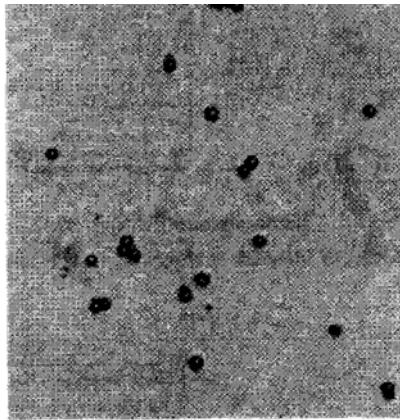


Fig. 14. Polystyrene particles prepared under standard conditions ( $\times 1200$ ).

bilizer was inhibiting coagulation between the particles, and making it possible to prepare the monodisperse particles. Also the increase of the concentrations of the media diluted the reaction system and retarded particle growth. The micrograph of the typical monodisperse polystyrene particles is shown in Fig. 14.

Polystyrene particles so prepared were used as contaminating particles on the substrate. Fig. 15 shows the typical micrograph of the particles deposited on a slide glass in our deposition chamber. The deposition occurred uniformly on the substrate and its edge effect on deposition was seldom observed. As shown in Fig. 16, the number of polystyrene particles deposited is proportional to the deposition time, which implies that the particle deposition occurs at a constant rate. Also the constant deposition rate means that the particles deposited previously do not affect the subsequent particle deposition. The effect of carrier gas flow rate on the particle deposition is shown in Fig. 17. As the carrier gas flow rate increases, the particle deposition increases up to 500 cc/min, flattens out, decreases to minimum around 1000 cc/min, and finally increases again. This variation would be related to the behavior of the particles near the surface. As the gas velocity increases, the rebound and entrainment of the particles increase in the laminar region, which



Fig. 15. Polystyrene particles deposited on the slide glass ( $\times 800$ ).

Room temperature=20 °C

Substrate temperature=25 °C

Baffle-to-substrate distance=15 cm

Slurry concentration=0.014 wt% PS in ethanol+water

Carrier gas flow rate=1000 cc/min

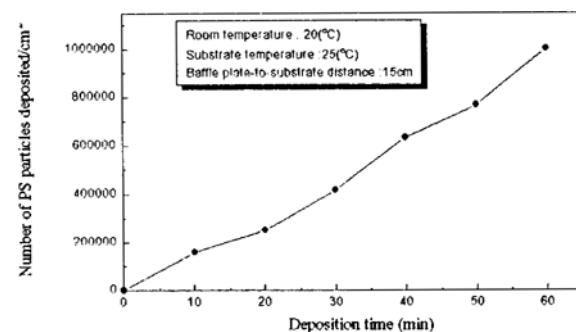


Fig. 16. Number of polystyrene particles deposited vs. deposition time.

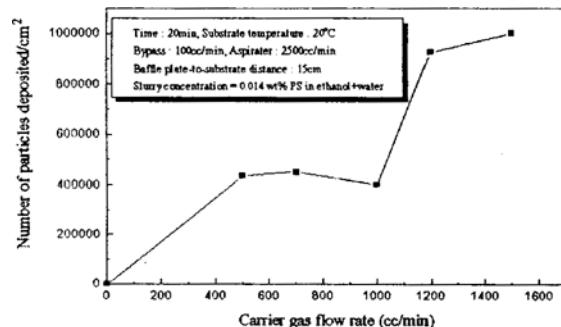


Fig. 17. Number of polystyrene particles deposited vs. carrier gas flow rate.

results in flattening and even decreasing particle deposition. However, as the velocity increases further, the deposition regime comes into that of turbulent deposition and the deposition efficiency again increases. In modeling particle deposition on surfaces, the particles are assumed to be transported by convection, Brownian motion, and external forces [Ye et al., 1991]. The external forces include gravity, thermophoretic and electrostatic forces. The particles having diameters far above 1 micrometer are transported largely by gravity while submicron

particles move mainly by diffusion. In contrast to these two particle transport mechanisms, thermophoretic velocity is almost independent of particle size. Therefore, its importance depends on the ratios of the thermophoretic velocity to the size-dependent velocities, such as the gravitational settling and diffusion velocities, respectively. For our particles having diameter of 2 micrometers, the relative importance of the three mechanisms of the particle transport is in the order of gravity>thermophoresis>diffusion [Hinds, 1982]. Fig. 18 shows the effect of the substrate temperature on the number of polystyrene particles deposited. As the substrate temperature increases above the ambient temperature, the number of particles deposited continuously decreases, while zero deposition is impossible to achieve due to the effect of gravity. Our preliminary numerical study [Shin, 1990] shows that 0.1-micrometer particles whose gravitational settling velocity is negligible, the effect of thermophoresis on the particle deposition would be appreciable and makes "particle-free" zone near the surface [Hinds, 1982]. The study also shows that for very small particles having diameter much less than 0.1 micrometer, the thermophoretic effect loses its dominance due to the diffusional effect, leaving no particle-free zone near the heated surface.

### 3. Removal of Particles

The % removal increases exponentially with time, as shown in Fig. 19. Since the rate of film removal is constant, the rate of particle removal is proportional to the number of the particles remained on it,  $n$ ,  $dn/dt = -kn$ , which gives the  $n/n_0 = \exp(-kt)$ , where  $n_0$  is the initial number of particles on the surface. Fig. 20 shows the effect of chamber pressure on the % removal of particles. The shape of the curve is different from that of the film removal due to the exponential nature of the % removal as

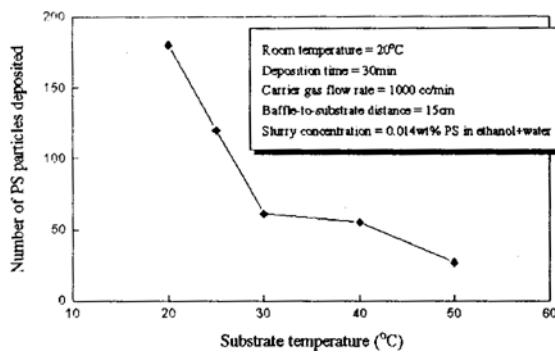


Fig. 18. Number of PS particles vs. substrate temperature.

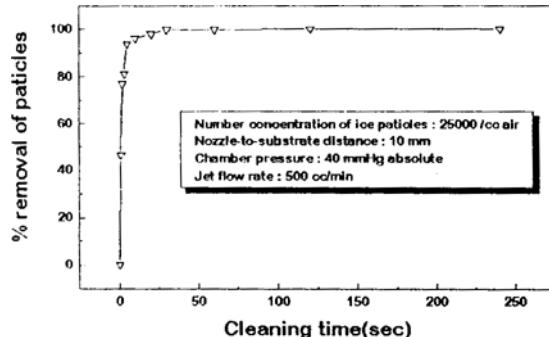


Fig. 19. % removal of particles vs. cleaning time.

just described. However, the critical pressure for obtaining the sonic velocity at the nozzle throat is again important in completely removing the particles deposited. Fig. 21 shows that the % removal of particles decreases as carrier gas flow rate decreases. At lowered velocity of the carrier gas with keeping the pressure of the cleaning chamber constant, the pressure of the ice-forming chamber, the lower part of the cleaning chamber, decreases correspondingly, so the pressure ratio decreases below the critical ratio. Subsequently, the nozzle velocity decreases below sonic, which resulted in the particle jet with the insufficient kinetic energy to clean the surface. The effect of the nozzle-to-substrate distance on the % removal of particles is shown in Fig. 22. The % removal decreases monotonically down to 30 mm due to the exponential nature of the particle removal. If the distance exceeds 30 mm, the removal of the par-

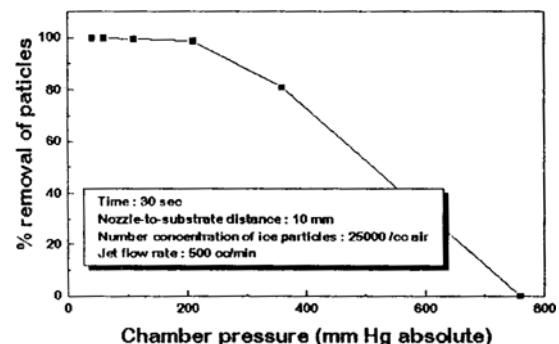


Fig. 20. % removal of polystyrene particles vs. chamber pressure.

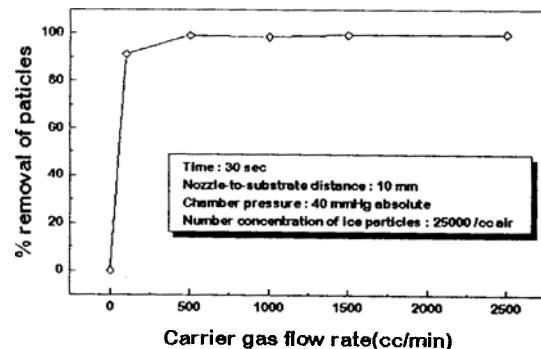


Fig. 21. % removal of polystyrene particles vs. carrier gas flow rate.

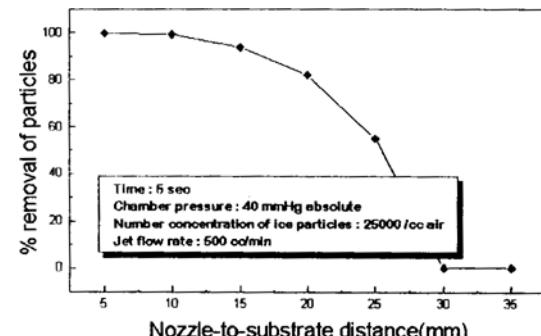


Fig. 22. % removal of polystyrene particles vs. nozzle-to-substrate distance.

ticles on the substrate has not been observed.

## CONCLUSION

The surface cleaning method has been developed using high speed ice-particle jet. The organic films and the surface contaminated with fine polystyrene particles were prepared as sources to be cleaned. The films include the lacquer film on the slide glass and the photoresist film on the wafer. Monodispersed polystyrene particles having average diameters ranging from 0.5 to 3 micrometers were prepared by changing the concentrations of the media. In deposition of the PS particles on a substrate surface, the parameters, such as the deposition time, carrier gas flow rate, and substrate temperature, controlled deposition efficiency of the particles. The removal of both the films and the contaminating particles was affected by the removal time, chamber pressure, carrier gas flow rate, particle number concentration and nozzle-to-substrate distance. When the ice particles have insufficient kinetic energy and/or the film to be removed has hardness higher than a critical value, the removal of film is retarded until the film gets mature for break. The method of ice-particle jet cleaning will have wide applications to removal of both the film and particles adhered on a surface, regardless of hardness of the film or adhesive force of the particles, since its cleaning power is purely physical and easily controllable.

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