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Experimental and Modelling Investigation of Re-Adhesion Mechanism of Detached Nanoparticles to Wafer Surface in Spin Rinse Process

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Adhesive particles on polished wafers are detached by e.g. scrubbing, and removed by liquid flow from the wafer. However, the relationship between the characteristic of liquid flow and the removal of detached particles has not yet been examined in detail. Therefore, the re-adhesion of detached particles to wafer surfaces in liquid flow on rotating wafers was experimentally investigated. The number of residue particles on a wafer was counted using a defect inspection tool after de-ionized water (DIW) rinse for particle removal. To discuss the mechanism of particle removal, a model was constructed and compared with experimental results. The model is based on boundary layer theory of fluid dynamics and advection diffusion theory of transport phenomena for particle removal in liquid flow near the wafer surface. The model results confirmed that detached particles in liquid flow moved into the sublayer and re-adhered to the wafer surface by diffusion. Moreover, particles in the sublayer, where the liquid velocity is several hundred um/s, could not be moved from the sublayer and removed from the wafer. It was also found that the number of particles re-adhering to the wafer surface depends on the Sherwood number.
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The technique of particle removal in liquid flow influences the productivity of chemical mechanical polishing (CMP) process, which is one of the semiconductor manufacturing processes, as shown in Fig. 1. CMP is composed of polishing, cleaning, and spin drying processes. The slurry abrasive size used in the CMP process is less than 100 nm. However, many abrasive and other material particles adhere to wafer surface after polishing. It is necessary to remove these particles from the wafer because residual particles on the wafer cause low productivity. As shown in Fig. 2, there are two steps in the CMP cleaning process. First, adhesive particles on wafer are detached by e.g. a polyvinyl acetal (PVA) Roll Brush.[1–3] The detaching process of particles from the wafer has been observed using an evanescent technique.[4,5] The contact conditions between the substrate and the PVA brush has also been investigated.[6] This technique is essential to understand interfacial phenomena such as polishing or cleaning of wafers. Second, the detached particles are removed by liquid flow from the wafer. Here, it is necessary to prevent the detached particles from re-adhering to the wafer surface. The zeta potential between the surface of the wafer and the particle is generally controlled to prevent the re-adhesion of particles.[7–9] It is also necessary to optimize liquid flow to prevent particle reattachment. The effects of wafer rotating speed and liquid flow rate on the liquid flow behavior have been visualized and reported.[10–12] The velocity of the liquid film surface on the rotating wafer was measured using imaging analysis and compared with the calculated value.[10–12] The liquid velocity near the solid surface is typically low due to viscosity. Therefore, when detached particles move to the wafer surface vicinity, particles cannot be removed, as shown in Fig. 2. However, re-adhesion mechanism of detached particles in spin rinse process is yet to be extensively examined.

In this study, re-adhesion mechanism was fundamentally investigated under designed experiment different with the real post CMP cleaning. Rinse would include various factors such as kinetic energy, pressure and diffusion. In this experiment, effect of diffusion on re-adhesion was fundamentally investigated under the designed experimental condition. To assume rinse process after scrubbing wafer, SiO_2 particle suspension was vertically supplied to a rotating wafer after supply of DIW was stopped, as shown in Fig. 3. Therefore, there is DIW between wafer and particle suspension when diffusion is ignored, as illustrated by step 2 in Fig. 7. The SiO_2 particle suspension was removed by DIW liquid flow. The number of particles on the wafer was counted using a defect inspection tool after spin-drying. Moreover, to investigate the mechanism of detached particles re-adhesion to the wafer surface, a physical model was constructed and compared with the experimental results. This model is based on the boundary layer theory of fluid dynamics and the advection diffusion theory of transport phenomena.[13–15] Comparing this model with the experimental results, the effect of the wafer rotating speed on the detached particles re-adhesion to the wafer surface is discussed in this paper.

Experimental

The removal characteristics of detached particles from the wafer surface, as shown in Fig. 2, were experimentally and fundamentally explored. An amount of liquid was vertically supplied to the center of a rotating Si wafer using cylindrical pipes. The pipe has an internal diameter of 4 mm. The Si wafer diameter is 300 mm. First, the entire wafer surface was covered with DIW supplied to the rotating Si wafer, as shown in Fig. 3. Second, after the supply of DIW was stopped, 50 nm SiO_2 particle suspension was supplied to a rotating wafer for 5 s. Finally, after the supply of the 50 nm SiO_2 particle suspension was stopped, DIW was supplied for 30 s to remove the SiO_2 particle suspension. Furthermore, after the supply of DIW was stopped, the wafer rotating speed was raised to 1000 rpm and the wafer surface was dried. The number of particles on the wafer was counted using a defect inspection machine (KLA-Tencor, Surfscan SP3). The difference in the number of particles before and after the experiment was calculated for each experimental condition. To investigate the effect of wafer rotating speed on particle removal characteristics, the particle removal experiments were performed under two conditions in which the wafer rotating speed was changed, as illustrated in Table I. To investigate the characteristics of particle adhesion to wafer surface, the wafer rotating speed was changed when SiO_2 particle suspension was supplied. Similarly, to investigate particle removal characteristics, the wafer rotating speed was changed when DIW was supplied to remove particles.

Results

Consequently, the number of residual particles per unit area on each wafer radial position is shown in Fig. 4. It was observed that the number of residual particles was greater when the particle suspension was supplied to a wafer rotating at a higher speed. On the other hand, the number of residual particles was smaller when DIW was

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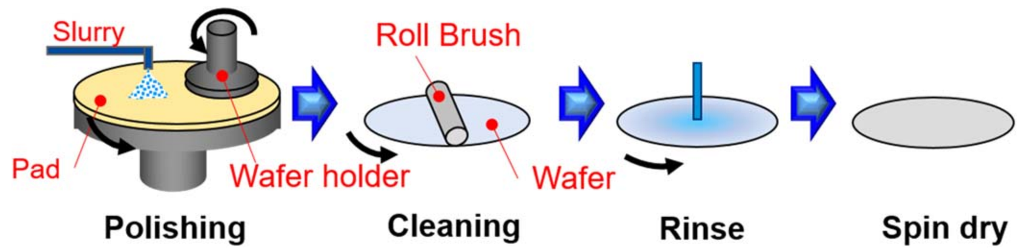


Figure 1. Schematic of CMP process.

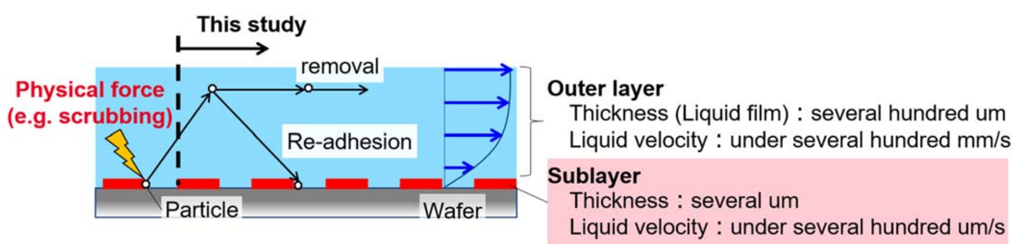


Figure 2. Particle removal in post CMP cleaning process.

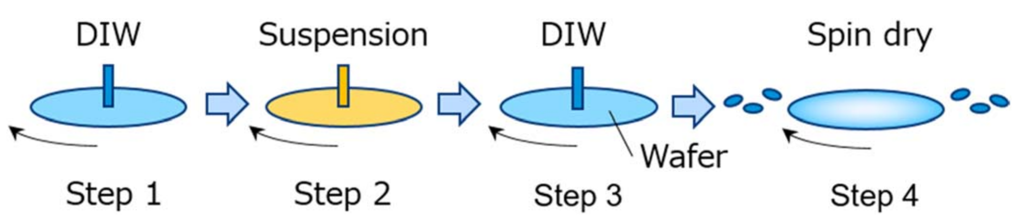


Figure 3. Schematic image of rinse experimental procedure.

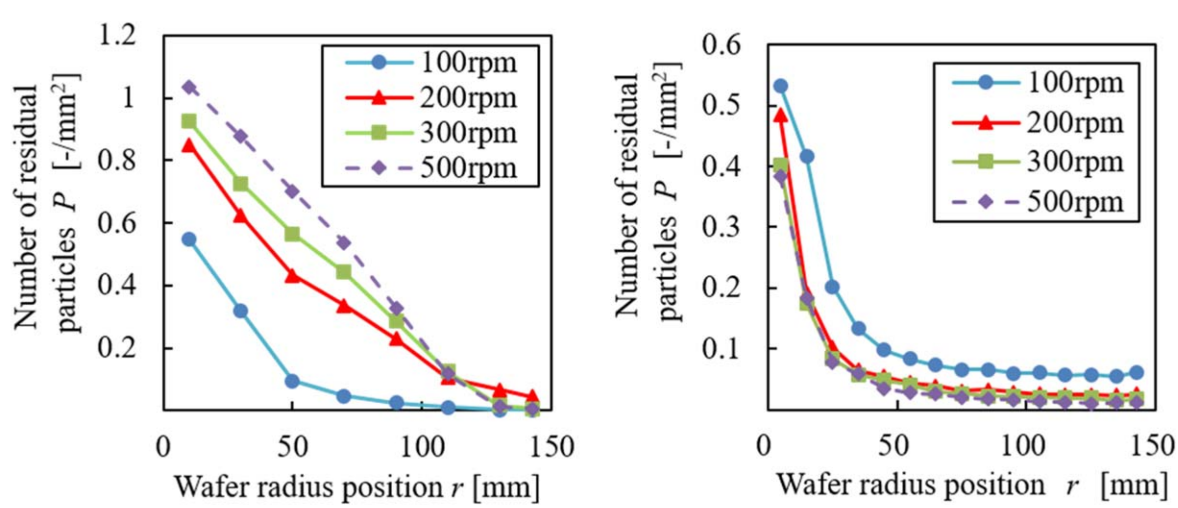


Figure 4. Experimental results regarding effect of wafer rotating speed for (a) Wafer rotating speed is changed when particle suspension is supplied and (b) Wafer rotating speed is changed when DIW is supplied for particle removal.

Table I. Experimental condition.

		STEP 1	STEP 2	STEP 3
①	Wafer rotating speed [rpm]	100	100, 200, 300, 500	100
②			100	100, 200, 300, 500
	Liquid type	DIW	Suspension (50 nm SiO_2)	DIW
	Flow rate [L/min]	1	1	1
	Process Time [s]	5	5	30

supplied to a wafer rotating at a higher speed for particle removal. The number of residuals particles on a wafer decreased in the wafer radial direction.

Discussion

Models to remove detached particles from wafer surfaces were developed using estimated liquid velocity distribution. First, a model based on the boundary layer theory of fluid dynamics was compared with experimental results. Second, a model based on the boundary layer theory of fluid dynamics and the advection diffusion theory of transport phenomena was compared with the experiment. Furthermore, comparing the model with the experimental results, the mechanism for the detached particles re-adhesion to wafer surfaces in liquid flow on rotating wafers is discussed in this paper.

Boundary layer theory model.—The liquid velocity distribution on a rotating wafer is calculated from the balance of centrifugal force and viscous force.[10]

$$-\mu\frac{\partial^2 u}{\partial z^2} = \rho r\omega^2 \quad [1]$$

Where, μ is viscosity, u is liquid velocity, z is distance from the wafer surface, ρ is liquid density, r is wafer radius position, and ω is angular velocity. Under the following boundary conditions, $u = 0$ at $z = 0$, $\partial u/\partial z = 0$ at $z = h$. Substituting these boundary conditions into Eq. 1

$$u = \frac{1}{\mu}\left(-\frac{1}{2}\rho r\omega^2 z^2 + \rho r\omega^2 Hz\right) \quad [2]$$

From continuity equation,

$$q = 2\pi r\int_0^H u \cdot dz = \frac{2\pi r \cdot \rho r\omega^2 H^3}{3\mu} \quad [3]$$

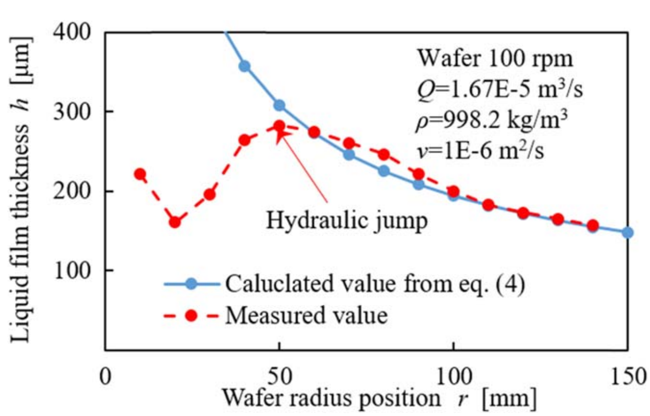


Figure 5. Comparison between estimation and measurement regarding liquid film thickness on a rotating wafer.

Liquid film thickness H on wafer radi position r could be obtained using the following equation.

$$H = \left(\frac{3\mu q}{2\pi\rho\omega^2 r^2}\right)^{1/3} \quad [4]$$

Where, q is liquid flow rate. The liquid film thickness from Eq. 4 is compared with the measured value as shown in Fig. 5. The liquid film thickness is from 200 to 300 um by centrifugal force. The measured liquid film thickness decreased after it increased around wafer radial position $r = 50$ mm. This phenomenon is called hydraulic jump and is not considered in Eq. 4. At each wafer radial position, the liquid velocity distribution obtained by Eq. 2 is shown in Fig. 6. The liquid velocity at the liquid film surface is several hundred mm/s, but the liquid velocity near the wafer surface ($z = 1$ um) is several mm/s. Therefore, from this calculation result, the particles near the wafer surface will be difficult to remove in the rinse process.

The liquid replacement process in the experiment is assumed, as shown in the schematic image in Fig. 7. The liquid is assumed to flow according to the liquid velocity distribution obtained by the boundary layer theory. The sectional image of the SiO_2 particle suspension flow is illustrated by step 2 in Fig. 7. In addition, step 3 in Fig. 7 indicates that the SiO_2 particle suspension is removed by DIW. Furthermore, using a fluid analysis software "FLUENT R19.2," the liquid flowed in the region where the wafer radial position $r = 70$–100 mm was analyzed under the assumption that the gas-liquid interface was a sliding wall. However, when a disturbance equivalent to 10% of the liquid velocity was given at the inflow entrance, the turbulence was dumped. It is assumed that diffusion and mixing had minimal flow near the wafer surface. Therefore, the number of particles in the suspension phase, whose film thickness is δ, should remain on the wafer after the DIW rinse. The thickness of particle suspension (δ) was calculated in the wafer radial direction. Hence, under these assumptions, the model that particles were removed according to the boundary layer theory without particle diffusion was developed and called the "boundary layer theory model" in this paper. The calculation process of the particle suspension thickness in the wafer radial direction is shown below. Liquid velocity $u(t)$ is written as the following equation.

$$u = dr/dt \quad [5]$$

Using Eq. 2 and 5,

$$\frac{dr}{dt} = \frac{1}{\mu}\left(-\frac{1}{2}\rho r\omega^2 z^2 + \rho r\omega^2 Hz\right) \quad [6]$$

When elapsed time (t) is 0 s, particle suspension starts to flow into $r = r_0$ and gets wet and spreads. r_0 is set as the diameter inside the nozzle. The moving distance of the liquid interface between the DIW and the particle suspension from $t = 0$ to $t = t_1$ can be calculated by the following integral equation.

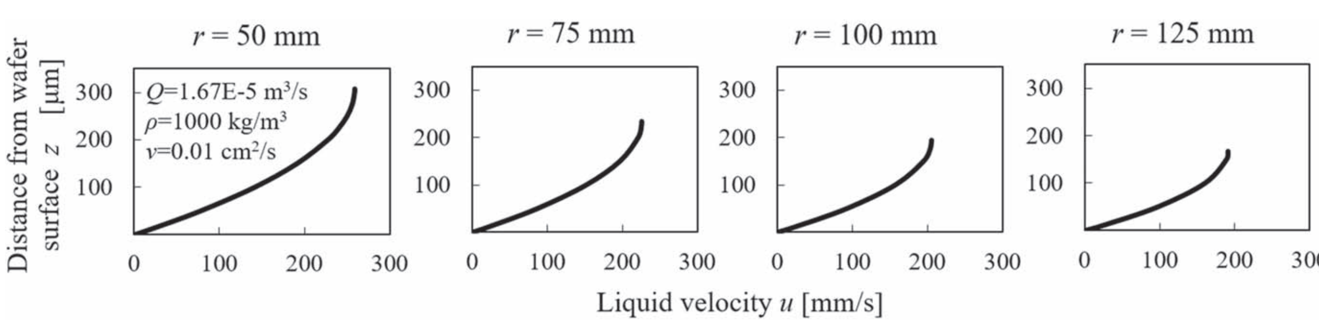


Figure 6. Liquid velocity distribution on a rotating wafer obtained by Eqs. 2 and 4.

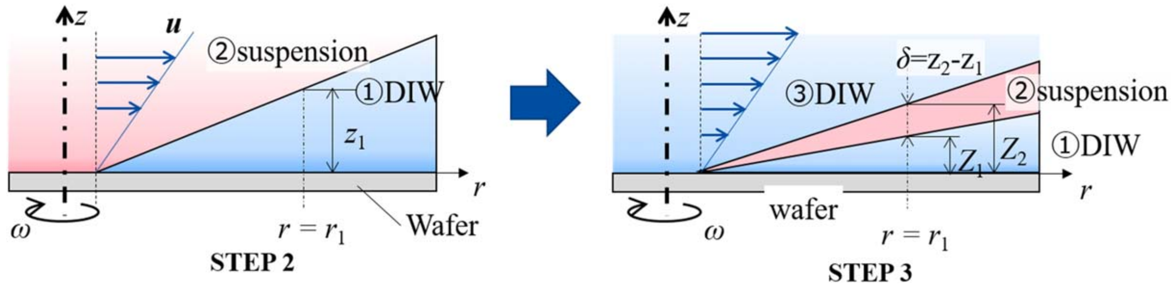


Figure 7. Schematic image of liquid flow process in experiment.

$$\int_{r_0}^{r_1} \frac{1}{r} dr = \frac{1}{\mu}\left(-\frac{1}{2}\rho\omega^2 z^2 + \rho\omega^2 Hz\right) \int_0^{t_1} dt \qquad [7]$$

$$r_1 = \exp\left[\frac{1}{\mu}\left(-\frac{1}{2}\rho\omega^2 z^2 + \rho\omega^2 Hz\right) t_1 + \log r_0\right] \qquad [8]$$

When the particle suspension is supplied to a rotating wafer, the conditions for existence of a liquid interface between the particle suspension and the DIW at $r = r_1$ are calculated. Equation 8 is arranged to a quadratic function of z, as shown in Eq. 9. For the existing liquid interface between the particle suspension and DIW, Eq. 9 has a real number solution.

$$z^2 - 2hz \dotplus \frac{2\mu}{\rho\omega^2 t_1} \log \frac{r_1}{r_0} = 0 \qquad [9]$$

When a discriminant is positive, Eq. 9 has a real number solution.

$$t_1 > \frac{2\mu}{\rho\omega^2 h^2} \log \frac{r_1}{r_0} \qquad [10]$$

Equation 10 gives the condition that the particle suspension flow into DIW on $r = r_1$. Using quadratic Eq. 9, the height of the liquid interface between the DIW and particle suspension z_1 can be obtained using the following equation.

$$z_1 = h - \sqrt{h^2 - \frac{2\mu}{\rho\omega^2 t_1} \log \frac{r_1}{r_0}} \qquad [11]$$

Moreover, when DIW is supplied to a rotating wafer for particle suspension removal, the height of the liquid interface between ②particle suspension and ①DIW z_1 is calculated in Eq. 12. Equation 12 can be obtained by the same process as from Eq. 5 to 9. The left side of the formula is the moving distance of the liquid. The first term on the right side is the moving distance when the particle suspension is supplied for t_1 seconds. The second term on the right side is the moving distance when DIW is supplied from t_1 to t_2 seconds for particle removal.

$$\int_{r_0}^{r} \frac{1}{r} dr = \int_0^{t_1} \frac{1}{\mu}\left(-\frac{1}{2}\rho\omega_1^2 z^2 + \rho\omega_1^2 h_1 z\right) dt + \int_{t_2}^{t_1} \frac{1}{\mu}\left(-\frac{1}{2}\rho\omega_2^2 z^2 + \rho\omega_2^2 h_2 z\right) dt \qquad [12]$$

The height of the liquid interface between particle suspension and the DIW for particle removal (z_2) can be calculated using Eq. 11. Using Eqs. 11 and 12, the thickness of the particle suspension ($\delta = z_2 - z_1$) can be calculated when the supply of DIW to remove particle is completed. It is assumed that particles in micro volume with thickness δ remain on the wafer. In reality, after the supply of DIW is stopped, the liquid on the wafer is blown away by centrifugal force and the wafer is dried. Therefore, the particle on the wafer might be removed by liquid flow during the spin-dry process. Here, the possibility to remove particle during the spin-dry process was estimated. Initially, temporal changes in the liquid film thickness (h_3) after the supply of DIW was stopped were calculated. The amount of discharge at the wafer edge should be the same as the integral value of the liquid velocity distribution Eq. 2 in the thickness direction. In addition, the amount of liquid discharge at the wafer edge multiplies the wafer area and the temporal changes in the liquid film thickness. When the liquid film thickness in the wafer radial direction is assumed to be constant as the thickness at the wafer edge ($h_{r\,=\,150\text{ mm}}$), the following Eq. 13 is obtained by the continuity equation.

$$-\frac{d}{dt}(\pi R^2 h) = 2\pi R \int_0^h u dz \qquad [13]$$

Where, R is the wafer radius. Temporal changes in liquid film thickness during the spin dry process h_3 is obtained by the following Eq. 14.

$$h_3 = \sqrt{\frac{1}{\frac{4\omega_3^2}{3\nu} t_3 + \frac{1}{h_0^2}}} \qquad [14]$$

Where, h_0 is the liquid film thickness at the wafer edge when the supply of DIW is stopped, ν is kinematic viscosity, ω_3 is angular velocity during spin-drying, and t_3 is elapsed time of spin-drying. Temporal changes in liquid film thickness h_3 during the spin-dry process are shown in Fig. 8. Furthermore, within several seconds after the spin-drying process starts, the liquid film thickness decreases to several micrometers and remains almost constant. There is almost no difference in wafer rotating speed. The calculation result of the number of residual particles is shown in Fig. 9. It was found that particles cannot be removed completely but remained on the wafer surface after spin-drying process. However, liquid film evaporates more rapidly than the estimation by Eq. 14, because evaporation is ignored in Eq. 14. Particles cannot be removed during the spin dry process. Therefore, in this boundary layer model, it is assumed that particles in micro volume whose thickness is δ remain on the wafer when the supply of DIW is stopped.

The boundary layer model is compared with the experiment results in Fig. 10. The solid line in the graph represents the analysis results from the model. The dotted line in the graph represents the experimental results. In Fig. 10-(a), when the particle suspension is supplied to a wafer rotating at a higher speed, the number of residual particles on the wafer becomes larger. The model results show the same tendency as the experimental results regarding wafer rotating speed. It is assumed that the particle suspension thickness δ becomes large since the height of the liquid interface z_1 is small when the particle suspension is supplied to a wafer rotating at a higher speed.

In Fig. 10-(b), when DIW was supplied to a wafer rotating at a higher speed for particle removal, the number of residual particles on the wafer was smaller. The model results show the same tendency as the experimental results for wafer rotating speed. It is assumed that the particle suspension thickness δ become smaller since the height

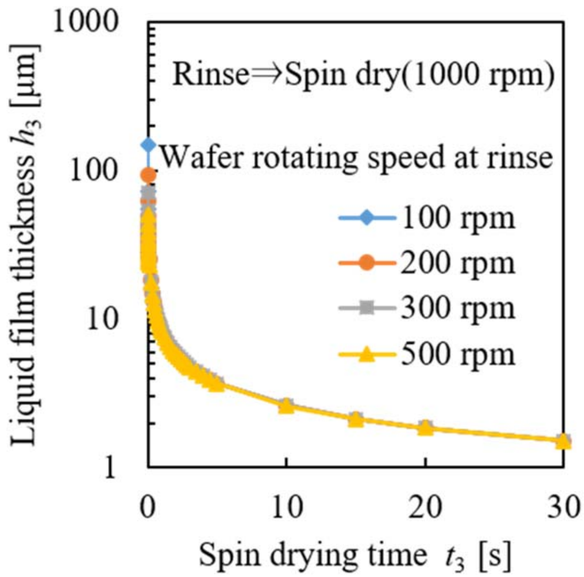


Figure 8. Temporal changes in liquid film thickness in spin dry process obtained by Eq. 14.

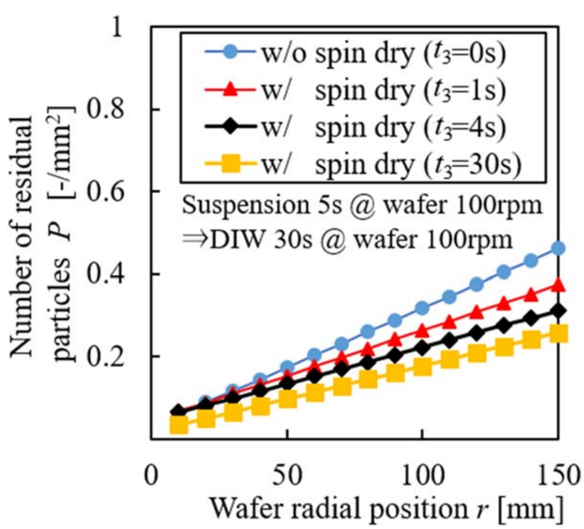


Figure 9. Estimation of temporal change in the number of residual particles on wafer.

of the liquid interface z_2 is smaller when DIW is supplied to a wafer rotating at a higher speed. However, there is a difference between the model and the experiment in respect to the characteristics of the residual particles in the wafer radial direction. The difference between the model and the experiment results because the particle diffusion effect is neglected. Moreover, Fig. 11 shows the change of the liquid interface height z_1 with time on each wafer radial position $r_1 = 50$ mm, 100 mm, 145 mm. It is assumed that particles on the central side of the wafer could approach the vicinity of the wafer surface since z_1 is smaller at the central side of the wafer. More particles on the central side of the wafer move to the vicinity of the wafer surface and remained on the wafer due to diffusion. Therefore, it is necessary to consider the effect of particle diffusion to remove particle by liquid flow on a rotating wafer.

Advection diffusion model.—To investigate the effect of diffusion on particle re-adhesion to a wafer, the unsteady advection diffusion equation was solved using the different method. As shown in Fig. 12, the particle removal process in liquid flow near the wafer surface is simplified. The liquid flow with velocity gradient is

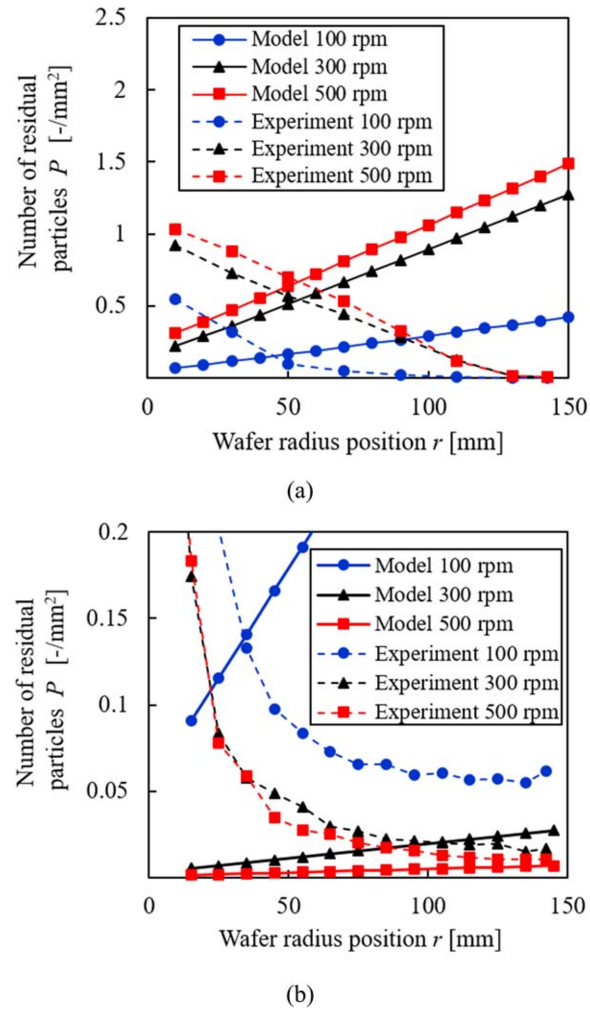


Figure 10. Comparison of experimental result and boundary layer model for (a) Wafer rotating speed is changed when particle suspension is supplied and (b) Wafer rotating speed is changed when DIW is supplied for particle removal.

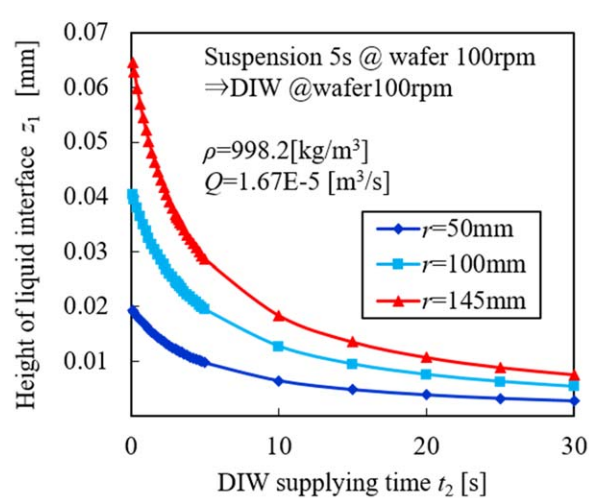


Figure 11. Temporal change in height of liquid interface between DIW and particle suspension (z_1).

averaged in the liquid film thickness direction and assumed to be the only one-dimension flow in wafer radial direction. In particular, the liquid velocity in the region where the distance from the wafer

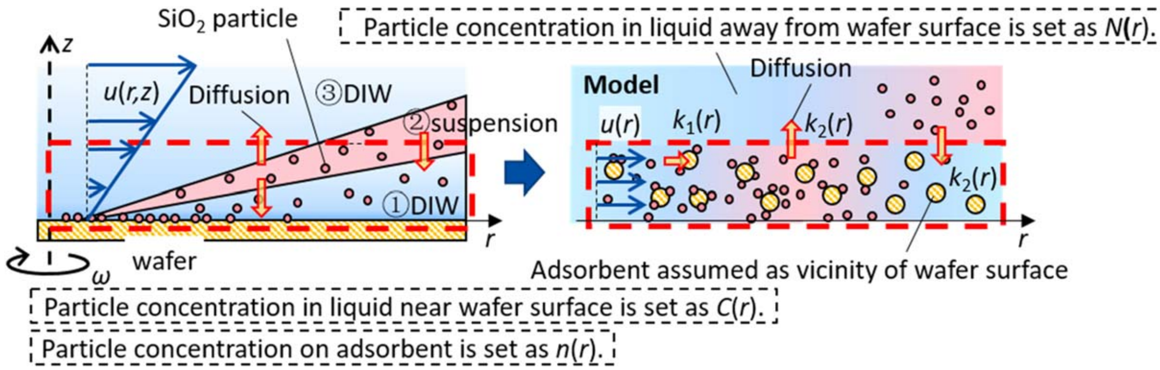


Figure 12. Schematic image of advection diffusion model for particle removal in liquid flow on a rotating wafer.

surface is within 100 nm is very small, zero point several mm/s estimated by Eq. 2. Particles near the wafer surface could not be removed during the rinse process because they remained in the region where the liquid velocity is small. In this model, mass transfer between liquid and the wafer surface is replaced by mass transfer between the liquid and the adsorption material fixed in the liquid. The adsorption material is set as the vicinity of the wafer surface. The particle concentration of the adsorption material is set as n. The particle concentration of the liquid flow near the wafer surface is set as C. The particle concentration of the liquid away from the wafer surface is set as N. The amount of mass transfer is determined on the basis of the Sherwood number, which is a dimensionless number used in mass transfer operations. This model is referred to as the linear driving force (LDF) model, which is used for simulation of component separation in chromatography.[13–15]

Furthermore, using the assumptions above, the advection-diffusion equation of the cylinder coordinate system is shown in Eq. 15. Where u is the mean liquid velocity in the region, and the distance from the wafer surface is several um. The first term on the left side is the unsteady term of particle concentration in liquid near the wafer surface; the second term on the left side is the advection term; the first and the second terms on the right side are the diffusion terms; the third term and the fourth term on the right side represent mass transfer between each region, as shown in Eq. 16 and 17. Advection diffusion equations are solved using a differential method, as shown from Eq. 15 to 17. The number of residual particles on the wafer is assumed to be the sum of particles in the liquid and the adsorption material. In this advection diffusion model, it is assumed that particles remain on the wafer when the supply of DIW is stopped, as particles cannot be removed in the spin-drying process, as shown in Fig. 9.

$$\frac{\partial C}{\partial t} + u\frac{\partial C}{\partial r} = D\left(\frac{\partial^2 C}{\partial r^2} + \frac{1}{r}\frac{\partial C}{\partial r}\right) - \frac{\partial n}{\partial t} - \frac{\partial N}{\partial t} \qquad [15]$$

$$\frac{\partial n}{\partial t} = D\left(\frac{\partial^2 n}{\partial r^2} + \frac{1}{r}\frac{\partial n}{\partial r}\right) + k_1(C - n) \qquad [16]$$

$$\frac{\partial N}{\partial t} = k_2(C - N) \qquad [17]$$

Where, k_1 and k_2 are mass transfer capacity coefficients which are obtained by dividing the film mass transfer coefficient k by the boundary film thickness. Where the boundary film thickness is set as z which is the height of the liquid interface obtained by Eqs. 11 or 12. The boundary film thickness is the region where molecular diffusion is dominant. The film mass transfer coefficient k is determined on the basis of the Sherwood number in parallel flow on the plane plate, using Eq. 18.[13] The Sherwood number is a complex function of temperature. The average temperature of the solution is about 293 K in this experiment. When the solution temperature rises by 5 K, the Sherwood number increases about 1%. The solution temperature T is set as 293 K, and the advection diffusion equation is solved.

$$Sh = 0.332 \cdot Sc^{1/3}Re^{1/2} = \frac{k \cdot r}{D}\left(Sc = \frac{\mu}{D},\ Re = \frac{ur}{\nu}\right) \qquad [18]$$

Where r is characteristic length, which is the wafer radial position. ν is kinematic viscosity. D is the diffusion coefficient and is determined from the Einstein Diffusion equation ($=k_BT/6\pi\mu a$). k_B is Boltzmann's constant, and a is the radius of a spherical particle. However, it was reported that the diffusion coefficient becomes smaller when the distance between the surface of a particle and the solid surface is reduced.[16,17] This is caused by an increase in apparent viscosity, as fluid movement is suppressed near the solid surface. It is deduced that the diffusion flux from the bulk of the liquid to the vicinity of the wafer surface is different from the diffusion flux from the vicinity of the wafer surface to the bulk of the liquid, because the particle diffusion coefficient is different. This depends on the distance between a particle and a solid surface. Furthermore, to investigate the difference in mass transfer direction above, this advection diffusion model was solved under following two conditions: case 1 is the condition that mass transfer between the bulk of the liquid and the wafer surface is interactive; case 2 is the condition that mass transfer is only one direction, from the bulk of the liquid to the wafer surface. The analysis model compared with the experimental result is shown in Fig. 13, when the particle suspension was removed by DIW supplied for 30 s. The analysis results of the interactive transfer condition show that almost all

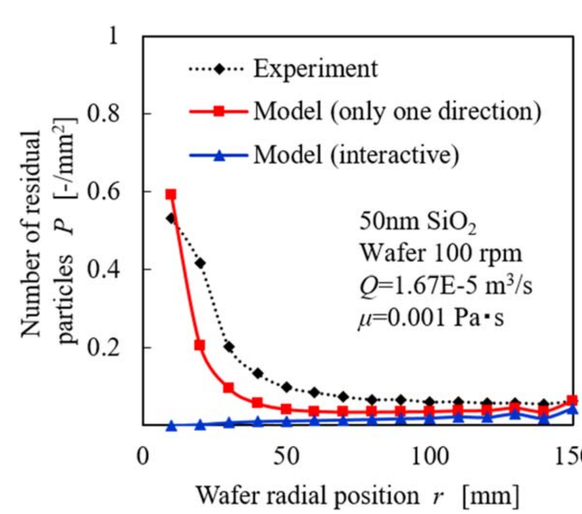
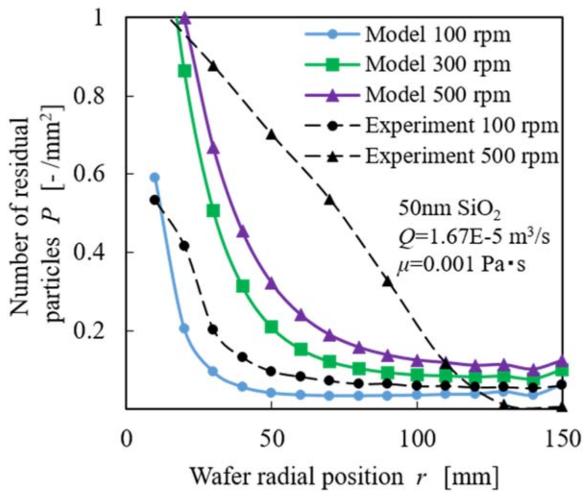
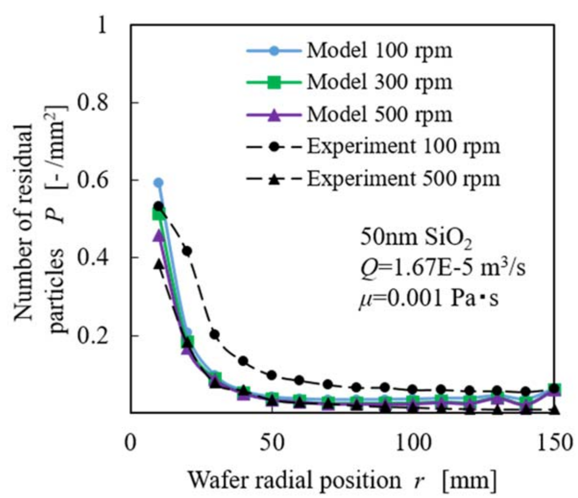


Figure 13. Comparison of the advection diffusion model for particle removal with the experimental results.



(a)



(b)

Figure 14. Comparison of advection diffusion model and experimental result for (a) Wafer rotating speed is changed when particle suspension is supplied and (b) Wafer rotating speed is changed when DIW for particle removal is supplied.

particles were removed completely, as shown in Fig. 13. This analysis result differs from the experimental result. On the contrary, the analysis result of a transfer condition in only one direction shows the same tendency as the experimental result. In this experimental condition, it is assumed that particles in the bulk of the liquid easily moved to the vicinity of the wafer surface in one direction by diffusion; however, particles in the vicinity of the wafer surface could not move to the bulk of the liquid. Therefore, particles were detached from the wafer surface.

Next, analysis results compared with experimental results for wafer rotating speed are shown in Fig. 14. The analysis results of residual particle characteristics in the wafer radial direction also show good agreement with the experiment. When the particle suspension liquid is supplied to a wafer rotating at a higher speed, more particles remain on the wafer as illustrated in Fig. 14a. It is assumed that particles move to the vicinity of the wafer surface from the bulk of the liquid, as mass transfer becomes higher when the wafer rotating speed is higher, according to Eq. 18. Thus, in regard to Fig. 14b, when DIW to remove particle is supplied to a wafer rotating at a higher speed, the number of residual particles on the wafer reduces. It is assumed that more particles move to the DIW phase, which is the gas-liquid interface side, as mass transfer increases when the wafer rotating speed is increased, according to Eq. 18.

Conclusions

In this study, the re-adhesion mechanism of detached particles to the wafer surface in spin rinse process was investigated. The number of residual particles on the wafer was counted after particle suspension on a rotating wafer was removed by DIW rinse. The effect of wafer rotating speed on the number of residual particles on the wafer was examined. As a result, it was found that the number of residual particles on the wafer becomes larger when particle suspension was supplied to a wafer rotating at a higher speed. To discuss re-adhesion of particles to a wafer surface, a model was constructed and compared with experimental results. The model was based on boundary layer theory of fluid dynamics and advection diffusion theory of transport phenomena for particle removal in liquid flow near the wafer surface. Consequently, the model significantly corresponded with the experiment. Therefore, it was confirmed that detached particles in liquid flow move into the sublayer and re-adhere to the wafer surface by diffusion. Moreover, re-adhesion of particles to the wafer surface cannot be moved out of the sublayer and removed from the wafer. It was also found that the number of re-adhesion particles to the wafer surface depends on the Sherwood number. These findings enable us to improve cleaning capability by controlling wafer rotating speed.

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