Protection schemes for critical surface in vacuum environments

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This article presents a study of particle formation during vacuum pump down and methods that can be incorporated to protect critical surfaces (like semiconductor wafers or masks) from those particles. Particle formation during pump down was reexamined with temperature measurements. Particles were intentionally produced with hard pump down for studying protection schemes. For the first step, a face-down approach for a critical surface was used to investigate the effect of protection from particle contamination. It was very effective to hold a critical surface face down for protection with the use of high gravitational settling velocity in vacuum environment. However, the face down approach did not sufficiently protect the critical surface. For the second step, a bottom protective plate was introduced below the critical surface to improve the protection efficiency. The bottom plate played a great role in protection of the critical surface with preventing particle formation near the critical surface by keeping the surrounding gas temperature high enough to avoid particle formation as well as with potential blocking of incoming particles toward the critical surface. Higher gas temperature intrinsically avoids formation of residue particles by the condensation process during pump down. © 2005 American Vacuum Society. [DOI: 10.1116/1.1978890]

I. INTRODUCTION

In many semiconductor manufacturing processes, vacuum systems and automatic operations are commonly used in order to minimize particulate contamination between processing steps and to satisfy other process needs. However, particles are frequently generated in various vacuum equipments, and the particles become a significant problem on improving production yields for manufacturers. Vacuum loadlocks, chemical reactions, and pump down are common sources of particulate contaminants in manufacturing processes from the sudden changes of manufacturing environment and system nonideality. In vacuum loadlocks, the transition period between normal pressure and high vacuum is the most significant contamination source on a wafer surface. In chemical reactions, unwanted by-products from many steps like implantation, plasma etching, metallization, and chemical vapor deposition are often generated within the system. In the pump down process, a large number of residue particles are generated by condensation under supersaturation condition from rapid adiabatic expansion of gas.

Particle formation during pump down was first reported by Hoh with witness wafers inside a chamber. Later, Strasser, Bader, and Bader published a result of reduction of particle contamination by controlled venting and pumping of vacuum loadlocks. Their approach to reduce particle generation during pumping is to stay in the nonturbulent flow regime. Chen and Hackwood reported that moisture is a necessary condition and turbulence promotes the nucleation, as independently described by Zhao. Zhao proved that particles are formed mainly by the supersaturation caused by sudden adiabatic cooling during pump down. In the studies of Zhao and Ye, Liu, and Pui, studies there was no particle formation observed when dry nitrogen was used for purging prior to pump down. Ye, Liu, and Pui did more systematic investigations for the residue particle formation during vacuum pump down and suggested three major steps involved in the residue particle formation: (1) absorption of SO2 and H2O2 into the condensed water droplets, (2) concentration of SO2 and H2O2 in the liquid droplets, and (3) oxidation of SO2 and H2O2 to form H2SO4. Ye also reported the mean size of the residue particles on various pumping
speeds. Mean sizes of residue particles ranged from 0.1 to 0.25 μm depending on pumping speed, and the smaller particles were formed in higher pumping speed.

At either humid or dry condition, system pressure during pump down shows exponentially decaying behavior with pumping time. Pumping time constant τ is included in Eq. (1); τ is the characteristic pumping time for certain system volume with typical pumping speed. Faster pumping speed has shorter time constant for the same system volume

\[ P = P_0 \cdot \exp(-t/\tau), \]  
\[ \tau = V/S_e, \]

where \( t \)=pumping time; \( \tau \)=pumping time constant; \( V \) =pumping volume; and \( S_e \)=effective pumping speed.

Zhao" measured the pumping time constant for the particle formation by changing relative humidity and set a criterion for the clean pump condition. In his criterion, for the higher humidity, the longer pumping time constant is needed to achieve clean pumping condition because of the increased particle formation in higher humidity. Due to the nature of loadlock systems for loading and unloading of cassettes in a clean room, a chance of particle formation still exists if the clean room air leaks into process systems between steps. Therefore, it is recommended to have a long pumping time constant which can provide clean pumping conditions for humid gas. For example, the boundary between clean pumping zone and particle formation zone is about 4 s of humid gas. For example, the boundary between clean pumping zone and particle formation zone is about 4 s of \( \tau \) from Zhao's criterion at 40% relative humidity, so the longer than 4 s of \( \tau \) is desirable to achieve clean pump down.

In this article, we are studying protection schemes for a critical surface in vacuum. The first protective step considered is a critical surface hung face down. Current and future manufacturing equipment especially for the extreme ultraviolet lithography (EUVL) will use the critical surface (such as the active side of a wafer or mask) face down for protection because particles settle fast at high vacuum environment due to the significant decrease of the drag force. Settling velocity is simply obtained by equating the drag force with the gravitational force for \( Re < 1 \), as shown in Eq. (3).

\[ V_s = \frac{p_o d^2 g C}{18 \mu}, \]  
\[ Re = \frac{p_o V d}{\mu}, \]

where \( p_o \)=particle density; \( d \)=particle diameter; \( g \) =gravitational acceleration; \( C \)=slip correction factor; \( \mu \)=air viscosity; and \( p_o \)=air density.

Several calculations were performed to show the importance of system pressure for the settling velocity and it is shown in Fig. 1. For example, the settling velocity of 100 nm particles is six orders of magnitude higher at 1 mTorr than at atmospheric pressure (760 Torr). So, the direction of critical surface is an important factor to avoid particle contamination making use of gravitational settling.

The second protective step is to mount a bottom plate within a small distance from the face-down critical surface during pump down. In this approach, the bottom plate will play an important role not only for stopping particles approaching the critical surface but also for preventing particle formation near the critical surface by keeping the surrounding gas temperature high between the critical surface and the bottom plate. The bottom plate, with high heat capacity compared to gas, helps maintain high gas temperature by convective heat transfer during pump down, and more discussions will be found in the result section.

**II. EXPERIMENT**

Figure 2 shows the schematic diagram of the vacuum chamber. The chamber is cylindrical in shape (pumping volume: approximately 20 l) equipped with a turbo pump and a mechanical pump in series, in order to achieve system pressures less than 100 mTorr. The isolation gate valve between the turbo pump and the mechanical pump was opened during pumping for studying protection schemes. The center tube was either closed for pump-down experiment using witness wafers as shown in Fig. 2(a) or connected with a vacuum particle detector (VPD, TSI 7340, lower detectable size limit \(-0.2 \mu m\)) for residue particle counts as shown in Fig. 2(b). During particle counts, the isolation gate valve was closed and the VPD associated with a remote processor (TSI 3704) was connected to a computer. After one cycle of pump down with the mechanical pump for the pressure down to 1 Torr, the chamber was pressurized by opening the purge air line. The purge air line includes high purity metal gas filter (Mott GasShield, removal of all particles down to 3 nm). The total number of particles formed during pump down with 40% relative humidity clean room air was measured. In examining protection schemes with witness wafers as shown in Fig. 2(a), two 6 in. wafers were placed one as face down attached to the chamber cover and one as face up on the bottom plate. System pressure is monitored with a MKS 226A pressure measurement system.
Using this chamber, we intentionally produced particles with the fastest pumping condition ($\tau \approx 1 \text{ s}$) during pump down, and those formed particles were used for deposition experiments. The shortest pumping time constant for the chamber system is approximately 1 s based on the system pumping volume of about 20 l, which needs approximately 7 s to achieve base vacuum. Different pumping time constant provides a different number of particles formed from the condensation process. We fixed 1 s of pumping time constant, which can provide the highest number of particles as shown in Table I, for investigation of protection schemes. In order to investigate the role of gap spaces between the critical surface and the bottom plate on protection, several different gap spaces were used. Spacers placed inside the chamber were used to support the bottom plate for each gap space: 0.5, 1.5, 3.7, and the 10 cm (which is effectively no protective cover plate). For particle deposition experiments, ten cycles of pump down were performed for particle formation, and then the wafer surfaces were scanned using a wafer scanner (PMS-3600, minimum particle size sensitivity of 0.1 $\mu m$ at 50% counting efficiency) after completion of pump downs.

An $E$-type thermocouple was installed to measure the change of temperature during pump-down process. The time constant of the thermocouple is approximately 0.1 s. The thermocouple was placed in the middle height inside the gap space and at the edge of the 7-in.-diam bottom plate, and it was replaced for temperature measurement of the chamber without the bottom plate 2 cm away from the chamber wall.

III. RESULTS AND DISCUSSION

Total particle counts measured with the vacuum particle detector during pump down are summarized in Table I for numerous pumping time constants. In Table I, there is clear boundary in total counts for the time constant between 3 and 5 s with three orders of magnitude higher in total counts. However, small numbers of particles were still observed up to 40 s of time constant. This measurement result is very similar to the prediction in Zhao’s criterion for clean pumping zone with the large number of total counts, but no complete agreement was shown for the small number of total counts. It is possibly because different system volume and different pumping system may produce different particle formation behavior. Particle formation is mainly due to the supersaturation by sudden adiabatic gas expansion, which provides cool gas temperature for condensation as also observed by Ye, Liu, and Pui.

Temperature change during pump-down was recorded for various gap spaces between the critical surface and the bottom plate. Figure 3 shows the results of temperature measurements, and these results imply that the bottom plate plays the role of a high heat capacity sink at close distance to the critical surface to keep gas temperature high. So, smaller gap space provides lower reduction of the gas temperature from adiabatic cooling effect because gas temperature is protected by convective heat transfer from the plate to the gas. Basic equations for pump-down process can be introduced to predict gas temperature change. The change of gas temperature is the result of the competition between adiabatic cooling and convective heat transfer from the chamber wall or the bottom plate which is surrounding the cool gas. So, the

![Diagram](image)

**TABLE I.** Particle counts using the vacuum particle detector (lower detectable size limit $\sim 0.2 \mu m$) with 40% relative humidity clean room air for numerous pumping time constants.

<table>
<thead>
<tr>
<th>Pumping time constant ($\tau$)</th>
<th>1</th>
<th>3</th>
<th>5</th>
<th>13</th>
<th>40</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total count</td>
<td>14500</td>
<td>12300</td>
<td>60</td>
<td>50</td>
<td>3</td>
<td>0</td>
</tr>
<tr>
<td>Prediction (Ref. 7)</td>
<td>Particle formation</td>
<td>No particle formation</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
gas temperature can be estimated by following mass and energy balance equations for the pump-down process

\[
\frac{dp}{dt} = -\rho \frac{S_x}{V} = -\frac{\rho}{\tau} \quad \text{(5)}
\]

\[
\frac{dT}{dt} = \frac{hA}{\rho C_V V} (T_0 - T) - \frac{(\gamma - 1)T}{\tau} \quad \text{(6)}
\]

with initial conditions of \(\rho = \rho_0, T = T_0\) at \(t = 0\), where
- \(\rho\) gas density;
- \(T\) gas temperature;
- \(h\) convective heat transfer coefficient;
- \(A\) surface area for the heat transfer;
- \(C_V\) heat capacity at constant volume;
- \(C_p\) heat capacity at constant pressure;
- \(V\) volume for the temperature measurement (gap volume);
- \(\gamma\) heat capacity ratio, \(C_p/C_v\).

Simple calculations were performed to estimate temperature variation during the pump-down process using the Eqs. (5) and (6) for different gap spaces. Figure 4 shows the calculation results from Eqs. (5) and (6). It clearly shows that the smaller gap space provides less reduction of gas temperature during the pump-down process. The model calculation was further verified qualitatively with the measured data in Fig. 4. Quantitative agreements between calculations and measurements are not completely perfect for the entire pumping process because of nonideality of the pumping process compared to the Eqs. (5) and (6). However, qualitative agreements of the temperature reduction for each gap cases are very good. Therefore, the high heat capacitive bottom plate plays an important role in preventing condensation particle formation during the initial pump-down process.

The minimum gas temperature was observed down to \(-38^\circ\text{C}\) for the pump down without using the bottom plate. Saturation ratio can be used to determine whether particle formation happens at the reduced gas temperature by the adiabatic expansion. The saturation ratio \(S_R\) is the ratio of the partial pressure of vapor in a system to the saturation vapor pressure for the temperature of the system \(1^\circ\text{C}\)

\[
S_R = \frac{p}{p_s} = \frac{2.34}{\exp\left(\frac{16.7 - 4060}{T - 37}\right)} \quad \text{(7)}
\]

where \(p\) and \(p_s\) are saturation vapor pressures of water vapor at 293 K (20 °C) and given temperature \(T\). The unit of the pressures is in kPa. This equation is only considered for water vapor condensation because it is the only source of initial droplet formation during pump down. We believe the composition of the final residue particles is H<sub>2</sub>SO<sub>4</sub> as reported by Ye, Liu, and Pui. \(^8\) Simple calculations were performed for the saturation ratio for the temperature range from 290 to 240 K in order to compare with our experimental observation. Table II shows the saturation ratio as well as critical saturation ratio obtained from the homogeneous nucleation.

**Table II. Saturation ratio of the water vapor in a system. System temperature is considered as 293 K (20 °C).**

<table>
<thead>
<tr>
<th>Gas temperature (K)</th>
<th>290</th>
<th>280</th>
<th>270</th>
<th>260</th>
<th>250</th>
<th>240</th>
</tr>
</thead>
<tbody>
<tr>
<td>Critical saturation ratio (^a)</td>
<td>3.2</td>
<td>3.6</td>
<td>4.2</td>
<td>4.8</td>
<td>5.5</td>
<td>6.6</td>
</tr>
<tr>
<td>Saturation ratio (^b)</td>
<td>1.2</td>
<td>2.4</td>
<td>4.8</td>
<td>10.5</td>
<td>24.8</td>
<td>63.4</td>
</tr>
</tbody>
</table>

\(^a\)Reference 8.
\(^b\)Reference 10.
Residue particles by condensation are expected to be most probable if the saturation ratio is higher than the critical saturation ratio. The saturation ratio is smaller than the critical saturation ratio for the temperature higher than 270 K (~3°C) in Table II, so the use of bottom plate with the gap space less than 3.7 cm is more than adequate to prevent particle formation from temperature measurements as shown in Fig. 3. In order to examine the effect of the bottom plate on protection schemes, we performed pump down with two witness wafers: face down and face up, and those wafers were scanned after ten cycles of pump down.

Scanned results for both face-down and face-up wafers without using the bottom plate are shown in Fig. 5 for the case of no bottom plate. It is evident that the pump-down cycle produces particles without the bottom plate, as explained above and in Fig. 3. It was believed that a face-down wafer would not contain any contaminants during pump down because gravitational settling is significantly increased at reduced pressures by decreasing drag force (increasing slip correction). Nevertheless, a face-down wafer still contained approximately 30 particles as shown in Fig. 5(a). The face-up wafer in Fig. 5(b) definitely had more contaminants (140) than the face-down one (30) because of the additional deposition due to gravitational settling. Settling speed increases significantly with decreasing system pressures as shown in Fig. 1 (pressure changed from atmospheric to low pressure during experiment was not considered in calculations). For the deposition on the face-down wafer, we believe those particles were deposited by transient flow behavior created during pumping as well as diffusional behavior. Therefore, the face-down wafer itself is not sufficiently protected from any particle sources, which can exist inside the manufacturing instrument. Consequently, we introduce the second concept of using the bottom plate for protecting the critical surface.

As mentioned above, the bottom plate intrinsically helps stopping the particle formation by keeping gas temperature high, so no particle deposition is expected with a bottom plate if the gap space is less than 3.7 cm. Scanned results from the 0.5 cm gap space case for both face-down and face-up witness wafers showed no particle additions in Fig. 6. Few particles on the two wafers were observed, but they are believed to be coming from the original blank wafers. Therefore, it is clear that the use of the bottom plate plays a crucial role in protection of the critical surface by comparing the results of 10 and 0.5 cm gap spaces. It is because the bottom plate intrinsically prevents particle formation from the rapid cooling of the gas during adiabatic expansion within the gap space. In addition, it is partly because the bottom plate protects any particles approaching the critical surface by transient flow behavior created during pumping.

So, the use of the bottom protective plate as well as the critical surface face down is considered as an excellent candidate for particle protection during pump down.

IV. CONCLUSIONS

We have investigated protection schemes for particulate contamination in a vacuum environment with witness wafers during pump down. Residue particles formed during pump down were used for examining protection schemes for the critical surface. The face-down surface is much more effective in avoiding particle contamination from the gravitational settling than the face-up case. Further consideration for the use of the bottom plate is promising to keep critical surface clean in addition to the face-down approach. The bottom plate plays a crucial role in not only avoiding particle formation by keeping the gas temperature high between the critical surface and the bottom plate but also preventing particles from approaching the critical surface.

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