PROBLEM

It is critical that the remote plasma sources used in semiconductor device processing be operated in a manner that contributes minimal particle accumulations per wafer pass on the substrates being processed. Appropriate equipment handling and conditioning, as well as optimal process procedures are necessary to maintain low particle contributions from these remote plasma sources. This application note describes the current best practices to minimize particle contributions when installing and using MKS Instruments’ R\textsuperscript{*} evolution III remote plasma sources in O\textsubscript{2}/N\textsubscript{2} ashing processes.

BACKGROUND

R\textsuperscript{*} evolution\textsuperscript{®} III

The R\textsuperscript{*} evolution III, an integrated remote plasma source with improved performance over the earlier R\textsuperscript{*} evolution I model, generates and delivers activated species required in a variety of semiconductor processes. Reactive gas generators are used in semiconductor processes such as photoresist ashing and thin film nitridation and oxidation.

The plasma chamber within the R\textsuperscript{*} evolution III remote source is a toroidal confinement vessel fabricated from a semiconductor-grade fused quartz, a non-crystalline high purity form of silicon dioxide (Figure 1).

Quartz – with its chemical inertness and excellent thermal and mechanical properties, has been adopted for multiple vacuum chamber applications in IC manufacturing. Quartz is also advantageous for plasma applications as it has a low recombination rate - 100 to 1000 times lower than on most metals and dielectrics, which results in higher reactive gas outputs for the R\textsuperscript{*} evolution III as compared with other remote plasma systems\textsuperscript{1}.

In-Process Particle Generation in the R\textsuperscript{*} evolution\textsuperscript{®} III

As is the case for all semiconductor processing equipment and components, low particulate operation is a critically important characteristic for these reactive gas generators. Within the R\textsuperscript{*} evolution III, a potential source of particle contamination is the inner torus surface. Over very long timescales, running O\textsubscript{2}/N\textsubscript{2} ashing recipes, the cumulative effect of occasional and transient plasma-quartz interactions within the torus can produce a redistributed form of silicon dioxide contamination we will hereinafter refer to as “condensed silica”.

Condensed silica contamination in these systems has two primary sources:

- **Plasma instabilities**: Transient plasma instabilities can produce localized wall heating in the torus which can, in turn, lead to evaporation of the top few atomic layers of quartz, forming condensed silica.
- **Ion Bombardment**: A very small number of plasma-generated ions can bombard the quartz surface, resulting in sputtering of the quartz surface to produce condensed silica.

**Plasma Instabilities**: During the transition between process recipe steps, the pressure, gas flow and/or RF power change while the plasma remains on. Under such conditions, the plasma may experience transient instabilities that result in direct plasma impingement on the internal quartz walls of the torus. This produces strong localized heating of the wall (Figure 2). This heating evaporates SiO\textsubscript{x} (x = 1 or 2) molecules from the quartz surface which are then entrained in the process gas flow. As the silica-laden process gas passes from the hot plasma zone to the cooler exit region of the quartz torus, gas phase SiO\textsubscript{x} molecules nucleate, forming condensed silica. Figure 3 shows a SEM image of the internal torus’ surface at the “cool” exit from the torus. The small, spherical-shaped features in the image are condensed silica that has accumulated on the interior torus walls after several hundred hours of RF operation. Figure 4 shows a comparable SEM image of an interior surface from the heated plasma zone of the torus after the same period of RF operation. The surface in Figure 4 was located about 10 mm upstream of the location of Figure 3. The lack of visible deposits in Figure 4 shows that the evaporated silica remains in the gas phase until it reaches the cooler post-plasma region.

\textsuperscript{1} http://www.mksinst.com/docs/R/toroidalTP.pdf
The formation of condensed silica due to plasma transients can be minimized by avoiding or eliminating plasma transients that produce the intense localized heat fluxes on the inner walls of the quartz torus.

**Ion Bombardment:** Plasma consists of a partially ionized gas created by high energy electron impact with gas molecules within a plasma. The RF field accelerates the relatively light electrons which impact ambient gas molecules. If the electrons have sufficient energy, the impact produces positive ions and additional free electrons. The lighter, much faster moving electrons quickly diffuse to the quartz walls producing a negative charge on the wall while the gas phase plasma is left with a positive charge relative to the wall. An electric potential thus develops in the dark space-charge region between the positive plasma and the negative quartz walls which is referred to as the plasma sheath region. Ions from the plasma that enter the plasma sheath are accelerated by the fields within. A very small percentage of these accelerated ions gain sufficient energy to traverse the plasma sheath and impinge on the quartz wall of the torus. These ions deposit their energy into the surface layers of the quartz; some (but not all) impact events release enough energy to sputter SiOx molecules from the surface. As with thermally evaporated silica, these molecules become entrained in the gas flow and subsequently nucleate and condense in the cooler downstream regions of the quartz torus.

Ion bombardment is inherent to the plasma generation and confinement geometry. Reducing the electrical energy in the plasma sheath will minimize but not totally eliminate the effect. The formation of condensed silica due to ion bombardment is minimized by using the lowest RF power necessary to achieve the desired process results.

**Photoresist Ashing**

The R* evolution III is primarily used for the removal of dry photoresist from wafer surfaces, a process commonly referred to as photoresist ashing.

Photoresist ashing processes employ a mixture of O2 and N2 typically in a 10:1 ratio. The simplest ashing recipes (Table 1) consist of two steps: a short gas flow stabilization step followed by an ignition/ashing step long enough to complete the ashing process. Figure 5 shows a typical process window for O2/N2 gas flows and chamber pressures. The RF power setpoint is a function of the pressure and flow as measured at the outlet flange of the R* evolution III and it is selected from a range of powers defined by a Process Power

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**Figure 2 - Cross-section A-A of the torus in Figure 1 showing: (a) a stable plasma; and (b) plasma impinging on the ID wall of the torus due to transient plasma instabilities**

**Figure 3 - SEM image (500x) of the internal surface at the exit to the quartz torus after several hundreds of hours of plasma operation**

**Figure 4 - SEM image (500x) of an internal surface of the quartz torus located within the plasma zone, 10 mm upstream from the site shown in Figure 3**
Setting Reference Chart\(^3\) for \(\text{O}_2/\text{N}_2\). As noted in the previous sections, the RF power setpoint should be as low as possible to minimize the formation of condensed silica. Differences between ashing chamber designs and pressure measurement location may impact the accuracy of pressure measurements at the \(\text{R*evolution III}\) outlet flange. To overcome any chamber specific issues, the previously referenced procedures establish the range of RF powers available for any final chamber configuration.

### SOLUTION

#### Process Recipe Optimization

Several process recipe strategies can help to minimize condensed silica formation in the \(\text{R*evolution III}\). These include:

- Low power operation
- Short recipe step times
- Turning the plasma off during recipe step transitions
- Recipe-step-transition smoothing while plasma-on

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#### Lowest power operation:

Empirical studies show a clear trend towards lower levels of particle adders with lower power operation (Figure 6). Lower power operation lowers the heat flux on the quartz and this, in turn, minimizes the generation of condensed silica.

![Figure 6 - Lower power setpoint reduces particle adders by \(\approx 10\times\)](image)

#### Short recipe step times:

Keeping the plasma-on step time(s) short minimizes the internal quartz temperature, reducing the generation of condensed silica.

#### Turning the plasma off during recipe step transitions:

If possible, a short no-plasma delay step should be employed between recipe steps that would otherwise produce significant flow, pressure or power set point changes.

#### Recipe-step-transition smoothing while plasma-on:

An alternative to the short no-plasma delay step is a series of 1 to 3 short plasma-on transition steps that effectively smooth out what would otherwise be an abrupt flow, pressure and/or power setpoint change.

### Hardware Installation and Foreign Material (FM) Contamination of Quartz Internal Surfaces

During \(\text{R*evolution III}\) chassis installation onto an OEM process tool, the user should take great care to avoid FM contamination of the internal quartz surfaces. While FM acts as a direct source of particulate contamination it can also act as a nucleation site for condensed silica from the process stream. FM typically originates from the local cleanroom environment, however, it may also derive from the VCR fittings that connect the process gas lines to the \(\text{R*evolution III}\).

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\(^3\) To create a chamber specific Process Power Setting Reference chart see the MKS Application note at: http://www.mksinst.com/docs/R/Revolution-ProcessPowerSetting-APPNOTE.pdf
The chassis is delivered with a sealed process gas inlet and a sealed process effluent exit flange to preserve the cleanliness of the internal surfaces. These seals should only be removed immediately preceding chassis installation. During chassis installation, extra care is necessary to prevent contamination from entering through these fittings as follows:\(^4\):

**Process Gas Inlet:** The male VCR threads on the R\(^*\)evolution III gas connection fitting and the female VCR threads on the OEM tool gas connection fitting should be thoroughly cleaned (using a cleanroom approved wipe or swab) prior to mating these fittings. In addition, the VCR glands should be held tightly aligned with the gasket in place while mating the female and male threads to prevent any particulate generation, due to the relative motion of the threaded parts, from infiltrating the process gas wetted path.

**Process Effluent Exit Flange:** The R\(^*\)evolution III weighs approx. 40 kg and should be installed with great care to avoid damaging the quartz torus located immediately adjacent to the aluminum exit flange. The alignment of the R\(^*\)evolution III exit flange, to its mating vacuum sealing surface on the OEM tool, should be guided by alignment pins or guides. Lowering of the R\(^*\)evolution III onto the vacuum sealing surface should be aided by a mechanical hoist or other mechanism. Human support of the full weight of the R\(^*\)evolution III during this procedure is strongly discouraged.

**Prevent Backstreaming of Contamination from the Ashing Chamber**

During system idle times or chamber vent, pumpdown or backfill times, or any other time when the possibility exists for ashing chamber contamination (particles) or condensable gases (photoresist solvents) to backstream into the R\(^*\)evolution III, a small flow of process gas (e.g., 500 – 1000 sccm of N\(_2\)) should be flowing to prevent such backstreaming.

Particles or condensates that have migrated up inside the R\(^*\)evolution can be released as particles at some later time or act as nucleation sites for the aforementioned condensed silica.

**Conditioning – Preparing the R\(^*\)evolution III for Low Particle Operation**

Fabrication, assembly, testing, shipping and installation onto an OEM process chamber of a R\(^*\)evolution III remote plasma source can expose the internal process-gas-wetted surfaces to ambient FM contamination. Such FM may be present as loosely adhered particles that are easily entrained in the process gas, producing subsequent particle contamination of the system. Alternatively, particle contamination may be present as FM that is strongly adhered to the wetted surfaces; such FM can act as nucleation sites for condensed silica which, in turn, can contribute to added particle counts. For this reason, a new R\(^*\)evolution III must be properly conditioned if it is to achieve optimal low particle operation.

Preparing the R\(^*\)evolution III for low particle operation requires a sequence of conditioning recipes with particle and/or contamination testing at various points in the sequence. Each R\(^*\)evolution III user should develop a conditioning recipe specific to their application. There are a number of guidelines that are useful in developing a conditioning recipe:

- **“Hi” or Aggressive Conditioning:** The first step of a typical conditioning sequence should be the performance of a more aggressive process recipe than that used in the standard ashing process (i.e., higher O\(_2\) and N\(_2\) flows at higher power and lower pressure). This will remove all but the most strongly adhered FM particles from the internal gas-wetted surfaces. Table 2 shows an example of such a recipe\(^5\). This aggressive treatment is repeated for as many as 200 cycles without removing the monitor wafer from the process chamber. Following completion of this step, the wafer is evaluated for added particles. The sequence is repeated, using a new wafer, until the added particles per cycle are below a pre-defined process specification, for example ≤ 20 adders per cycle, ≥ 90 nm\(^3\).

<table>
<thead>
<tr>
<th>Conditioning - Hi</th>
<th>Stabilize</th>
<th>Process</th>
<th>Idle</th>
</tr>
</thead>
<tbody>
<tr>
<td>O(_2) (sccm)</td>
<td>5500</td>
<td>5500</td>
<td>5500</td>
</tr>
<tr>
<td>N(_2) (sccm)</td>
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<td>550</td>
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<td>0</td>
</tr>
<tr>
<td>Time (sec)</td>
<td>10</td>
<td>120</td>
<td>60</td>
</tr>
</tbody>
</table>

Table 2 - Conditioning recipe “Hi”

\(^4\) It is recommended (per the R\(^*\)evolution III product definition drawing) to use gas filtration of > 6 LRV for particles > 0.003 μm at each process gas inlet.

\(^5\) Power set points in excess of the maximum power achievable for a given flow and pressure condition will result in the maximum power being delivered for that flow and pressure condition. A power warning message may also be seen on the LCD display.
• "Lo" or Process Conditioning: A second conditioning recipe should follow performance of the aggressive or "Hi" conditioning step. This recipe should employ O₂ and N₂ flows similar to those expected for the production ashing recipe but at lower power and higher pressure settings than in the "Hi" recipe. An example recipe is shown in Table 3.

Again, the conditioning step is run for as many as 200 cycles, without removing the monitor wafer from the process chamber. After this conditioning sequence, the wafer is evaluated for added particles. Such a sequence is repeated until the added particles per cycle are below a pre-defined process specification, for example ≤ 5 adders per cycle, ≥ 90nm.

Contamination testing, such as VPD-ICP/MS analysis, may be used in one or both of the conditioning sequence steps to monitor for specific contaminants of interest.

### CONCLUSION

Process recipes that use low power and short process times achieve the best performance for low in-process contamination in photoresist ashing recipes. The removal of RF power between recipe steps and/or customizing recipe step transitions to reduce or eliminate abrupt changes in flow, pressure and/or power can further optimize a process for low in-process particle generation. Conditioning of the internal quartz surfaces as well as the use of purge flows to prevent backstreaming of contaminants during idle and/or vent times will further contribute to low particle operation. In addition to these process protocols, strict compliance with proper cleanliness during R*evolution III chassis installation procedures will minimize the amount of FM contamination present in photoresist ashing systems.

Adherence to these process protocols and installation guidelines will optimize R*evolution III-equipped OEM photoresist asher for minimal particle adders during the initial process startup and qualification and help to maintain reliable particle performance for the life of unit.

### REFERENCES

For more information on MKS Instruments remote plasma sources, link to our web site:

For a general catalog of MKS remote plasma sources, follow this link:
http://www.mksinst.com/docs/UR/PlasmaSources-DS.pdf