Application Note 112

Moisture Behavior in Ultra-High Purity Gas Distribution Systems

Introduction

Modern semiconductor fabs employ sophisticated gas distribution systems (GDS) to deliver a variety of ultra high purity (UHP) gases from bulk purifiers to the process tools. These GDS are controlled and monitored by Continuous Quality Control (CQC) systems that allow fabs to maintain extremely low levels of contaminants in their process gases, leading to high process yields. The task of controlling contaminants is becoming more demanding as device line sizes decrease at an unprecedented rate and the impurity control points of UHP gases are reduced to their limits.

The principal contaminants monitored by CQC systems are oxygen and moisture. Of these, moisture is the most difficult to control due to its adsorption and transport characteristics, particularly at sub-ppb levels. These characteristics not only gate the initial dry down of a GDS but also make leak detection and process trouble shooting of process excursions difficult.

The purpose of this Application Note is to familiarize users of CQC systems with the difficulty of making ultra-trace level moisture measurements, to set a practical expectation for moisture analyzer performance and to establish guidelines for the proper use of these analyzers in controlling and monitoring moisture in UHP gases.

The following sections of this note will discuss leak mechanisms in gas distribution systems, moisture transport phenomena and behavior in GDS, the implications of these on making moisture measurements and guidelines for the proper use of ultra trace moisture analyzers in CQC systems.

Leak Mechanisms in Gas Distribution Systems

Leak mechanisms can be characterized in one of two ways:

Actual Leak - A direct leak path from atmosphere located directly on an actively flowing UHP line.

An a*ctual leak* usually produces a fixed rate of atmospheric contaminants entering the UHP system, which enter via back diffusion. Therefore, if the flow of UHP gas increases, the concentration of the contaminants will decrease proportionately due to the dilution effect of the higher UHP gas flow rate. Similarly, if the flow of UHP gas decreases, the concentration of contaminants in the UHP gas downstream of the leak source will increase overall.

Virtual Leak - A trapped pocket of contaminated UHP gas which is in a static flow condition (dead leg), but is in direct contact with the active gas distribution system

The trapped gas pocket can have an *actual leak* which is acting as the source of contamination, but that source by this definition is located some distance from the actively flowing UHP gas and is separated by the pocket of trapped gas. Under steady state flow conditions of the actively flowing UHP gas, the contamination is mostly contained within the dead leg. However, some smaller amounts of contamination will continuously diffuse out of the dead leg and into the UHP gas stream.

If the pressure of the flowing UHP gas drops slightly at the connection point to the trapped pocket of contaminated gas, then contaminated gas will flow out of the trapped space and into the main flow. In this case, a pressure drop in the flowing line is usually associated with an increase in flow (gas demand). Therefore, for a v*irtual leak*, if the flow of UHP gas increases, it will actually cause an increase in the concentration of contaminants momentarily until a new steady state (diffusion as the only mechanism spreading contamination into the UHP gas flow) can develop.

By the same token, if pressure were to increase slightly, clean UHP gas would rush into the trapped pocket which will work against the steady state diffusion of contamination into the UHP gas stream. Accordingly, a decrease in UHP gas flow will usually cause a momentary decrease in contaminant concentration in the UHP gas flowing downstream of the dead leg. Depending on the rate of contaminant diffusion from the trapped pocket and the flow rate of UHP gas, the decrease in measured concentration in the flowing gas when the diffusion is suppressed may be difficult to detect. More typically, pressure changes are only momentary as gas demand cycles change. This causes small amounts of contaminated gas from the dead leg to enter the flowing gas which would be detected downstream as somewhat diluted contaminant spikes.

There are many complex interactions of dynamic factors pertaining to a v*irtual leak*, such as the volume of trapped gas, its relative concentration of contaminants, whether the dead leg does or doesn't have an a*ctual leak* within it that is replenishing contaminants that diffuse or get purged out of the dead leg and into the bulk flow, etc. These interactions make repeatable and predictable measurements more difficult than with an a*ctual leak.*

Moisture Transport Phenomena & Dynamics

Moisture may be present in the GDS due to the presence of residual adsorbed moisture on the tubing stock used to fabricate the delivery system, to actual or virtual leaks as discussed in the previous section (i.e., back diffusion) or from a malfunction of the bulk purifiers. Residual moisture present in the system at startup is removed through long term purging of the system with UHP gas during the dry down (outgasing) cycle. Once eliminated, it is no longer a problem unless the system is inadvertently exposed to high levels of moisture, requiring another dry down cycle. Moisture due to leaks or purifier malfunction is eliminated by purging with UHP gas and may be done on-line or off-line depending on the degree of contamination. In either case, moisture reduction to the required process level can take a long time (hours to days) due to the nature of the water molecule.

Moisture is the most difficult impurity to remove from UHP systems because of its adsorption/desorption characteristics. Initially, moisture is chemisorbed onto the distribution tubing surface. Then, additional layers of moisture are physisorbed onto to base layer and tend to be in equilibrium with moisture in the boundary layer of the gas phase [1]. Elimination or reduction of either layer of moisture is difficult because of the length of time it takes for the adsorbed moisture to equilibrate and diffuse into the relatively dry gas flowing in the system (both are inherently slow and are rate-determining processes).

Several papers have been written about successful attempts to model moisture behavior in distribution systems and in system components (valves) based on chemical/physical models and provide insight into how slow the drydown process can be. In a paper by Dheandhanoo et al [2], the authors experimentally determined dry down times for various tube and pipe materials with different surface finishes. They present data for drydown of electropolished (EP) and non-EP stainless steel tubing as shown in Figure 1, below.

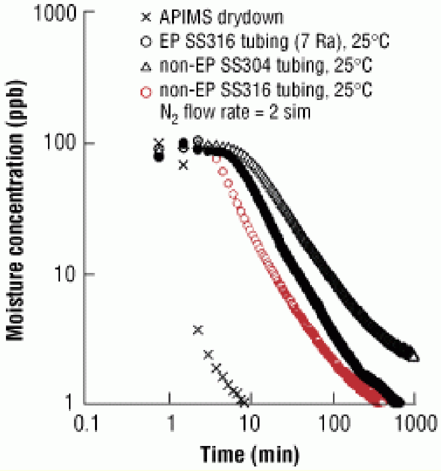

Figure 1. Dry-down characteristics EP SS316 (7 Ra), non-EP SS316, and non-EP SS304 tubing samples, all at 25°C with a 2 slm N2 flow rate, and the APIMS dry-down characteristics.

For EP SS316 tubing, dry down from 100ppb to 1ppb takes about 800 minutes or over 13 hours. Considering that the process control set point is usually much less than 1 ppb (typically 0.2ppb) and that atmospheric exposure could lead to a starting point well above 100ppb, dry down times can easily exceed days and more.

In an article by Funke et al [3], the dry down characteristics of diaphragm valves with different seat materials was examined. The graph below (Figure 2) shows the length of time it takes to drydown a diaphragm valve with different seat materials from just under 1000ppb to below 1ppb. At levels close to a process control set point of 0.2ppb, the dry down time can exceed 600 minutes or 10 hours. If the starting point is higher, the dry down is even longer.

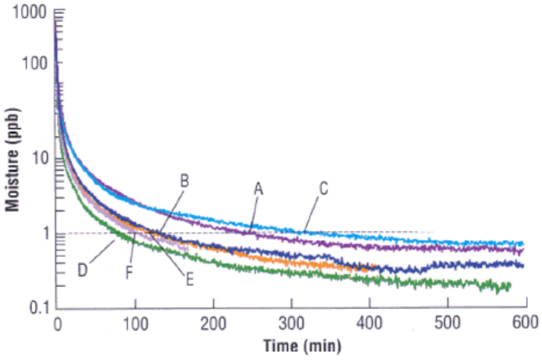

Figure 2. Comparison of polymer- and metal-seated valves at high moisture challenge. A) new Kel-F at room temperature (RT); B) Kel-F repeat at RT; C) new Kel-F at 70°C; D) dry Kel-F at 70°C (5 min open); E) metal at RT; and F) metal at 77°C.

In a paper by Ma and Verma [4] on 'Moisture Drydown in Ultra High Purity Oxygen Systems', the authors found that drydown of oxygen systems took considerably longer than drydown in inert gas systems due to recombination effects of dissociated moisture as H and OH radicals. In our experience, oxygen gas is also problematic after drydown, whether it's dealing with normal operation or zeroing, because the dynamic response of moisture in oxygen is much slower than in inert gases. So, it is not just the nature of moisture that affects drydown characteristics but the nature of the UHP gas as well.

In dealing with real GDS in fabs, it is our experience that significant and persistent moisture contamination can take weeks to months, in some cases, to clean up. This topic is explored in more detail in the following section.

Implications for the Use of UHP Moisture Analyzers

The experimental results discussed above show that moisture adsorption/desorption phenomena lead to long clean up times of UHP GDS compared to other contaminants, especially oxygen. (In fact, detection of oxygen is so easy compared to that of moisture that it is the preferred way to leak test GDS. For more details on this, refer to a companion piece, Application Note 106, *Non Invasive Leak Detection in UHP GDS*.) These phenomena also lead to slow response of changes in moisture levels within the GDS. A typical GDS system is shown in Figure 3. The system depicted is conceptually simple but can produce complex moisture profiles. Gas from the purifiers at the gas pad flows into two mains with multiple laterals off of each main. Each lateral may have multiple process tools connected to them or may not be populated at all. Furthermore, a lateral may or may not be active at any given time. Each tool, in turn, can be at any stage in its processing cycle.

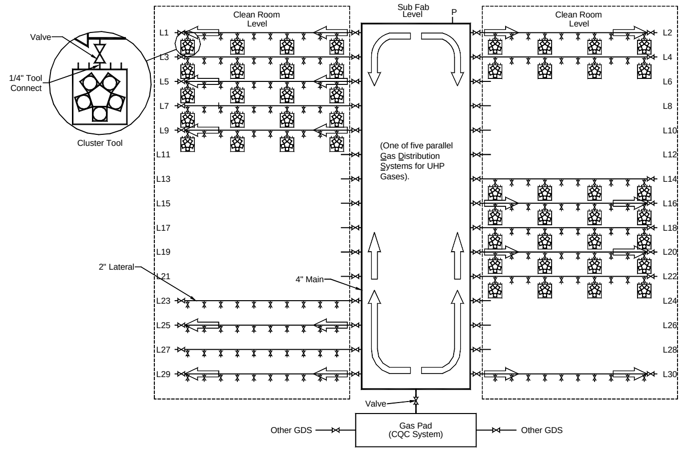

Figure 3. A Typical Gas Distribution System in a Semiconductor Fab

Flow and pressure changes within the system in conjunction with ambient temperature effects produce unsteady-state moisture profiles. The list of variables that affect moisture response within a GDS is long and varied, including:

- Materials of construction
- Tube diameter, length and surface finish
- GDS geometry, and component type and placement
- Temperature and pressure changes, ambient and internal
- Gas type, purity and flow rate
- Tool use and state (e.g., normal operation, maintenance mode, inactive)
- Back diffusion from process tools

The dynamic response of a moisture analyzer in a CQC is gated by all of these factors. The contribution of the analyzer to the overall system dynamic response is essentially nil compared to the GDS itself.

Because of the slow dynamic response of a distribution system, the amount of control 'head room' available is crucial to effective process control. Head room is defined as the amount of latitude between the impurity spec and the analyzer's low detection limit. The greater the amount of head room an operator has, the better the chance that he will be able to bring a wayward process back into control before a purity spec is violated. Ideally, it is recommended that a 10:1 ratio between the contamination specification for the particular analyte and the lowest detection limit (LDL) of the analyzer be used. Practically, the control ratio should be no lower than 5:1. This means that a gas contamination spec of 1ppb requires an analyzer with an LDL of 0.1ppb at best and at least 0.2ppb practically speaking.

Because of the complex geometry of a typical GDS and the effect of back diffusion from outside sources and from virtual leaks, the moisture trace can be unpredictable and difficult to interpret. This makes finding and explaining the root cause(s) of a disturbance very difficult if not impossible with any degree of confidence. Our experience is that first-time users of UHP analyzers can be overwhelmed by what they see and tend to blame the analyzer for the irregularity and not the process – the analyzer is 'guilty until proven innocent'. More often than not, the analyzer is correctly reporting the presence of moisture.

Guidelines for the Proper Use of Ultra Trace Analyzers in UHP Gas Systems

Note: The guidelines below are largely generic to any installation but some are product specific.

Installation

- Avoid system designs that allow large or rapid changes in the ambient temperature environment of both analyzers that are mounted in the CQC system and external GDS mains and laterals. For example, avoid having heated or cooled air blow directly on the analyzers or CQC cabinets, or on distribution system components.
- Minimize the length of the sample lines to minimize the effects of temperature changes (adsorption, desorption of moisture on the walls of the GDS tubing).
- Effective temperature control of the rack is paramount to effective moisture monitoring and control. In designing the air conditioning system for the CQC rack, be aware of the natural temperature gradient in the rack, with the analyzers highest in the rack running the hottest.
- The CQC cabinets must allow for the proper flow of air into the front of the instruments. This is easily accomplished by designing the cabinet so that air flows into the front of the rack and by providing enough space between the front of the instruments and the cabinet doors to allow for good circulation.
- Provide high quality power and grounding to the CQC system to avoid spurious electrical effects.
- Minimize the amount of ambient dust that the analyzers are subjected to to avoid dust buildup inside the analyzer. Check the filter element in the analyzer door to insure it is not clogged with dust and particulates. Clean as necessary. Likewise, check the air filter in the CQC cabinet to insure that it is clean as well.
- Provide reasonable access to instruments, particularly the rear of the instruments, for ease of maintenance and troubleshooting.
- Good CQC system design should include sample bypass lines to allow for fast initial drydown of the sample pathway and fast dynamic response during normal operation.
- Be aware of the potential for RFI problems from remote radio devices during start-up and routine operation.
- Insure that all sample lines to the analyzers are cleared of debris prior to startup. Make sure the sample line filter gaskets (frits) are installed and keep a supply of spare gaskets on hand.
- Locate the vacuum pump so that the potential effects of vibration and heat build up on the instruments are minimized.

Start Up

- Be aware of the length of time it takes to properly dry down the analyzers and the GDS system. The dry down time is not fixed but is affected by many variables; use moisture level end-point determination as a guide instead of elapsed time.
- The higher the moisture content of the gas sample, the greater the need for effective temperature control. This is especially true at start-up when the moisture content is typically high.
- Take full advantage of any operator training provided by the analyzer manufacture or system supplier. This is a small investment that will provide an enormous return given the size of the capital investment already made.

Zeroing

- During the first month of operation on process gas, do not zero the instrument. Use factory zero if at all possible after the first month; in most cases, it is a better quality zero than that generated by the user.
- If unit must be re-zeroed, allow ample time for drydown. Use the Active Zero feature to manage the baseline as the instrument and system continue to dry down.
- When initially installing the purifier on a DF-760 (dual oxygen and moisture analyzer), follow this procedure to minimize the amount of ambient air introduced to the moisture sensor:
  - Turn oxygen sensor off
  - Restore zero gas flow for the oxygen sensor only (while it is off)
  - Quickly install the purifier
  - Allow zero gas to flow through oxygen sensor for a few hours or overnight
  - Turn oxygen sensor on
  - Restore process gas flow through oxygen sensor

- Dry down the analyzers for several days using purified process gas as a means of showing that the analyzer is working properly. For best results, run the analyzer on purified gas for about 22 hours and then on process gas for 2 hours. This will build operator confidence in the instruments' readings and accelerate the purge down of the sampling system.

Leak Checking

- Application Note 106 provides a good primer on leak checking.

Good Operating Practices

- In case of doubt of the analyzer's reading, turn the purifier on to see if the readings go down and then return to the original value when turned back off. This approach is very good at building the operator's confidence in the unit and to demonstrate that the analyzer is tracking real moisture in the sample.
- If the operator is having to make frequent pressure adjustments to stay within the operating window, this could be a sign of slow pump wear and may signal the need for a pump rebuild (a simple, fast procedure). A simple test for pump wear is to isolate the analyzer with the pump on; if the pressure does not drop below 125 Torr, it is time to rebuild the pump.
- The lower the moisture control limits, the greater the need for good ambient temperature control.

References:

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