

DELTA F CORPORATION
APPLICATION NOTE NO.
106



Non-Invasive Leak Detection in Ultra-High Purity Gas Distribution Systems

Introduction

Ultra-High Purity (UHP) gases with bulk gas piping distribution systems are widely used in the semiconductor manufacturing industry. The requirement for faster, higher capacity semiconductor components is driving semiconductor process technology limits and specifications for impurities in UHP gases have been gradually dropping due to increased demands for purity in semiconductor fabrication processes.

UHP gases have been produced for years with virtually no contaminants (<0.1 ppb), but the challenge has been to deliver those gases at UHP levels all the way to the process tools. Improvements that have been made in recent years are reflected in the lower impurity level specifications now possible at the newest semiconductor fabs. Bulk gas distribution systems can be quite complex. Acceptable system integrity depends upon continuous perfect operation of numerous components within the distribution system that can provide a leak path from atmosphere, such as bad valve seats, diaphragm seals, or capped service taps accidentally left loose.

New semiconductor fabs typically require trace gas impurities at or under 1.0 ppb, whereas under 10 ppb would have been the limit in the early 1990's. UHP gases typically now have < 0.1 ppb of each contaminant as delivered to point-of-use. But, when used in real-life production environments, most discrete gas analyzers for use at ultra-low trace levels do not have a Lower Detection Limit (LDL) low enough to measure contaminant levels at the maximum impurity specification limits. As a result, an Atmospheric Pressure Ionization Mass Spectrometer (APIMS), with detection limits below 0.05 ppb, is usually required to qualify the integrity of a UHP gas distribution system. However, the capital and operating expense of an APIMS makes it a poor choice for continuous dedicated gas quality monitoring.

UHP gases and their distribution systems are very reliable and are always qualified at UHP levels prior to commissioning. However, these systems do break down and technicians inevitably make mistakes. The result can be a leak from atmosphere of moisture, oxygen, nitrogen, carbon dioxide, etc., that causes hundreds of thousands of dollars in scrapped wafers. The cost can be much higher if the contamination source is not identified and fixed quickly.

This Application Note explains how Oxygen Analysis can be used reliably and cost effectively to guarantee the continuous delivery integrity of all UHP bulk gases, except oxygen. The analysis information can be used to protect sensitive processes in real time by interrupting delivery of contaminated UHP gas, and further to help locate the leak. Moreover, oxygen analysis can be used without the need to shut down the entire system as is required by invasive leak detection methods, such as testing for leaks using a mass spectrometer with helium as a leak tracer gas.

What is the Value of Non-Invasive Leak Detection?

The common method of identifying leaks in a gas distribution system is, unfortunately, bad wafer product. Sometimes, the result of an atmospheric contamination can be seen visually, in the form of a hazy, dull surface finish (hazing) on the wafers caused by oxygen in an annealing or epitaxial process. Often, subtle contamination, such as uncontrolled thin oxide growth (<50 Angstroms) during a diffusion process, can go unnoticed because the effects are not detected until contaminated wafers reach a test step. By then, depending on the contamination and the process it affects, several production lots may need to be scrapped.

The classic approach to checking gas distribution system integrity is by the detection of helium using a Mass Spectrometer. This is done to verify integrity after initial fabrication, and after a process failure to locate the leak. The procedure requires helium to be flooded into the piping branch to be checked. Then a high sensitivity mass spectrometer detector is used to sniff around every joint, valve, and fitting for helium that may be leaking (outboard leak check). Alternatively, a hard vacuum is pulled on the piping branch and the mass spectrometer, and helium is flooded along the outside (inboard leak check). Both versions require that the piping system be taken out of service for some time during tests, and during the purge down of the system with UHP gas following the tests. Further, this method can only be used to verify system integrity under a static condition, and only on an occasional spot-check basis.

The "Non-Invasive Leak Detection" technique is a faster and easier way to identify an ambient atmospheric leak, or breach in the gas distribution system, without interrupting the normal gas supply. Further, because this approach does not continuous "real-time monitoring" basis. Unlike the invasive helium approach typically used after a catastrophe, the Non-Invasive approach can be used to identify a leak that is developing and, thereby, avoid an unscheduled shut-down. The key is to identify a suitable leak tracer gas that is not found in the UHP gases but that appears in the event of a leak. Preferably, this gas species should be detected before fabrication processes become corrupted.

Why Detect Oxygen When Processes are Sensitive to Other Contaminants?

Oxygen is a logical choice for monitoring on a continuous basis to identify a piping distribution system failure before tool processes are corrupted. Oxygen is normally at ultra-low trace levels in all UHP bulk gases. It is readily available in the atmosphere (20.9% O₂ compared to the 1-2 %H₂O typically found in climate controlled air). Oxygen is a highly mobile molecular species with favorable surface adsorption properties. In other words, it will find the leak path and readily diffuse into the UHP gas much faster than H₂O would. Once inside the distribution system, the poorer adhesion of oxygen to the inside surfaces of the piping insures that it will travel with the prevailing flow or diffuse rapidly to a sampling location where an analyzer can be detect its presence.

Lastly, there is now a practical and cost effective analytical tool sensitive enough to detect minute leaks in the realm of the Helium Mass Spectrometer's capability, but it can do so on-line in real time, without a disruption to normal UHP gas delivery. The Delta F ultra-low trace oxygen analyzer had proven performance of < 0.1 ppb sensitivity (smallest detectable change) and < 0.2 ppb LDL. In numerous tests of side-by-side comparisons to APIMS, the Delta F NanoTrace Analyzer has demonstrated analytical detection levels well below any other discrete gas analyzer of any type.

Of course, an APIMS has better performance specifications than the NanoTrace Analyzer, but it is neither practical nor cost effective for widespread monitoring. One APIMS system costs more to own and operate than 20 NanoTrace Analyzers. That translates into twenty times more sampling locations of continuous monitoring by using NanoTrace Analyzers, for the same cost as a single APIMS. Moisture analysis is not as effective to verify gas distribution system integrity as oxygen analysis is because of the performance characteristics of typical discrete moisture analyzers, and because of the properties of moisture itself.

Moisture prorogates into and through the distribution system much slower than oxygen does. Also, the moisture concentration from a spike of contamination will be reduced due to the moisture adsorption to the inside walls of the piping system. By the time the moisture reaches the sample tap, there is far less to be measured than is actually leaking in.

The NanoTrace Oxygen Analyzer responds several times faster and is much more sensitive than a discrete moisture analyzer, by nature. Its detection limit is nearly 20 times lower than typical UHP moisture analyzers. The fast response of the NanoTrace analyzer (90% of a step change in <20 seconds, typical) and its high sensitivity insures that even transients, like contamination event spikes, are detected. Therefore, given its speed and sensitivity, the NanoTrace analyzer is the best choice to discover and localize leaks, as well as to indicate when a leak has been successfully repaired.

How is Oxygen Monitoring Accomplished?

Most semiconductor fabs incorporate a multi-contaminant analysis system to verify levels of key impurities (i.e., H₂O, O₂, CO, CO₂, particles, and hydrocarbons) in each of the UHP bulk gases distributed within the fab. Due to cost constraints, this multi-component measurement is typically made only at one location – usually at the end of the main in the distribution system (exit purity).

Exit purity analysis is an absolute minimum to assure that UHP gas purity is being achieved in the fab. But what happens if a leak develops in a line that branches off of the main? Depending on gas usage patterns (which cause flow and pressure variations within the piping system), it is quite possible that the contamination would never reach the main line, or that a quick spike would propagate into the main line but be too brief for slower analyzers to detect. Additionally, a spike is likely to be diluted down to below the analyzers' detection limit. In either case, the contamination would never be detected by exit purity analyzers.

A better approach is to monitor more locations, but at less cost per point. This enables a more widespread protection of the entire distribution system, including out toward tool locations, at a lower overall cost than one complete multi-contaminant entrance or exit purity analysis system. The most common sources of contamination in bulk gas distribution systems are from accidental exposure, misconnection, or component failure. They almost always originate from an atmospheric exposure or cross-contamination. A careful study of the piping in the distribution system, along with the usage demands of the process tools and the locations of the tools that are most sensitive to atmospheric contamination will yield a map of potential locations for continuous monitoring. Select points which optimize between widespread protection of the entire piping system and monitoring gas feeds in close proximity to the more contamination-sensitive tools. This effort should yield a reasonably short list of locations for continuous monitoring in each UHP gas piping network.

Once the locations for continuous monitoring sample points has been determined, oxygen analyzers must be selected and installed. Analyzers should have a reliable lower detection limit (LDL) which is at least 2-5 times below the Oxygen Impurity Specification Limit for the UHP gas of interest. Ideally, a continuous data acquisition system should record data points at least once per minute, and have the capacity to record trending over a few weeks at a time.

Following installation and an initial equilibration (clean-up) period for the analyzers, each should be operated for several days on purified source gas to characterize its zero baseline performance. Chart the data with sufficient resolution to determine the Peak-to-Peak Noise, (band width of signal output while sampling oxygen-free "zero" gas), and zero baseline drift over a one-week period in order to validate the analyzer's performance. Then, compare the analyzer

trend data while sampling purified UHP gas to trend data taken while sampling the normal process UHP gas. This exercise will determine the "Contaminant Signature" (band width of typical baseline oxygen contamination at a particular location in the distribution system).

Provided the analyzer has an adequately low detection limit, comparison of trending data taken over time vs. the typical "Contaminant Signature" will uncover a potential leak well before risk to any fab production process has occurred. The trending data can be used to determine Warning Alarm setpoints, which can be set below the UHP gas product specification limits, but outside the "Contaminant Signature" range. An alarm trip at this level would prompt further investigation. A consequential breach of the distribution system will show up as an out-of-spec condition, but it will be caught early and readily localized by the network of online oxygen analyzers.

Leak Mechanisms

Leak Mechanisms can be characterized in one of two ways: Actual Leak – A direct leak path from atmosphere located on actively flowing UHP line.

An actual leak usually has a fixed rate of atmospheric contaminants entering the UHP system, which enter via diffusion. Therefore, if the flow of UHP gas increases, the concentration of the contaminants will decrease proportionately due to dilution effects 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.

Virtual Leak – A trapped pocket of contaminated UHP gas which is in a static flow condition (dead leg), but is adjoining to the active gas distribution system.

The trapped gas pocket can have an actual leak which is providing a 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 of UHP gas, the contamination is mostly contained within the dead leg. However, small amounts of contamination will continuously diffuse out of the dead leg and into the UHP gas flow.

If the pressure of the flowing UHP gas drops slightly at the connection point to the trapped pocket of contaminated gas, the contaminated gas will flow out of the trapped space and into the main flow. In the case, a pressure drop in the flowing line is usually associated with an increase in flow (gas demand). Therefore, for a virtual 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 can develop (in steady state diffusion is the only mechanism spreading contamination into the UHP gas flow).

By the same token, if pressure were to increase slightly, clean UHP gas would move into the trapped pocket and work against the steady state diffusion of 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 puffs 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 virtual leak, such as the volume of trapped gas, its concentration of contaminants, whether the dead leg does or doesn't have an actual leak that is replenishing contaminants that diffuse or get purged out of the dead leg, etc. These interactions make repeatable and predictable measurements more difficult than with an actual leak.

Leak Locating Within the Distribution System

Once a leak in the gas distribution system has been uncovered by the on-line oxygen analyzers, the leak source can be located by using an additional portable analyzer for spot-checking. First the data from the on-line analyzers can be correlated with data on pressure and flow changes within the system to identify a smaller area where the leak may reside. Analyzers that are not detecting abnormal oxygen levels are assured to be upstream of the leak. This will minimize the area of the system to be spot-checked.

In the absence of pressure and flow data, review the process tools' gas usage patterns. A tool cycle will cause local supply pressure to drop slightly when cycling on, and increase slightly when cycling off. The subtle pressure changes cause gas to flow which can either transport contaminated gas further into the distribution system, or suppress the spread of contamination by pushing back contaminated gas which is trying to diffuse into the distribution system.

The portable oxygen analyzer can be used next to sample from unused tap locations near, but downstream of the suspected leak. The closer the sample location is to the leak source, the higher the concentration will be. Therefore, spot-check measurements may be at higher concentration and can be made more quickly.

The unique Hand-Carry Portable configuration of the NanoTrace analyzer makes it an ideal tool for investigative, spot-checking measurements. The NanoTrace analyzer can be configured for Hand-Carry Portable use by selecting the Supplemental NICAD Battery Power, Isolation Valves, and On-Board Calibration options. With the flip of two diaphragm valves to isolate the sensor, the analyzer can be kept running while it is hand-carried from sample tap to sample tap. Upon reconnection, the NanoTrace analyzer is immediately ready to make accurate low ppb readings within minutes, providing the sample line and connection to the analyzer, and then allowed to purge further prior to tightening the last connection. This thorough pre-purging technique will virtually eliminate upsets from transporting, and can enable even sub-ppb readings to be made accurately within an hour.

Because of this Hand-Carry Portable capability, the NanoTrace can be used in inaccessible locations, where the typical MOP (Moisture, Oxygen, Particles) analytical carts cannot access, such as the gas jungle locations on tools. By using the pressure, flow, and/or gas usage techniques to deduce the location of the leak, it is possible to fix leaks before they lead to a process failure or shutdown.

What Makes Delta F So Different?

New standards have been set in UHP gas analysis with the introduction of the first practical analytical tool with detection limits approaching the normal impurity levels in UHP gases. The NanoTrace analyzer has a sensitivity of <0.1 ppb (smallest detectable change) and an LDL of <0.2 ppb. These performance levels were determined by several independent tests that show a near perfect correlation with an APIMS, when sampling oxygen step challenges of 0.1 to 1.0 ppb. This is nearly 10 times better than any other discrete oxygen analyzer.

The Nano Trace analyzer uses an extension of the well-proven Delta F Non-Depleting Coulometric sensor technology, which has given Delta F analyzers a reputation for years of reliable, drift-free operation. The Delta F sensor uses carbon-based electrodes which do not undergo chemical change to make measurements, hence the term Non-Depleting. The proprietary, inert carbon-based electrodes are driven by an external 1.3 VDC potential and extremely stable. Other ultra trace oxygen analyzers use Hersch type galvanic sensor technology. These sensors use a consumable cadmium anode electrode, which itself develops the electrochemical potential needed to drive the O₂ reactions, but it is inherently much less stable than the Delta F electrode.

Further, Delta F includes an exclusive system to the NanoTrace sensor for Electrolyte Conditioning. The Stablax™ System removes dissolved oxygen from the electrolyte without the need for sparging, or otherwise bubbling sample or zero gas through the electrolyte. Sparging (bubbles) introduces significant electrochemical noise to the sensor output preventing clean and predictable low detection levels. To ensure long term zero stability, the Nano Trace sensor also employs a patented technology to protect the sensor from the inevitable ionic impurities in the electrolyte. Other analyzers do not provide this level of protection from impurities. They require frequent calibration and electrolyte maintenance, resulting in excessive and unnecessary down-time.

Recognized for Quality

Delta F's Quality Management System has been certified to ISO-9001 by Lloyd's Register Quality Assurance Ltd. This audited compliance with an internationally accepted standard assures you of the highest quality in product design, manufacturing, and service.

Delta F Oxygen Analyzers can be ordered with a full scale range of 0-2 parts per billion (ppb) to as high as 0-25 percent. For specific product recommendations, contact Delta F Corporation, 4 Constitution Way, Woburn, MA 01801-1087, Tel. (781)935-4600, FAX (781)938-0531, e-mail marketing@delta-f.com.