APPLICATION NOTE

INFRARED TECHNOLOGY FOR CHAMBER CLEAN ENDPOINT DETECTION

PROBLEM

Chemical vapor deposition (CVD) processes deposit material on all the surfaces in a CVD process chamber, including the target substrate, CVD chamber walls, and internal component surfaces. Over time, the deposits on the internal surfaces in the chamber increase in thickness, to the point where small particles of the CVD material can delaminate, producing gas-phase particulates that deposit on the substrate and negatively impact process yield. Different chemical etching processes are available for remote and in situ plasma chamber cleaning that can be used to periodically remove deposits from chamber walls thereby maintaining chamber cleanliness. These processes require a method for detecting the endpoint of the clean to ensure that only the CVD deposits are removed, with no etch damage done to the chamber and component surfaces. Over-cleaning can produce surface damage that reduces chamber component lifetime and increases baseline particulate levels, significantly increasing system cost of ownership.

BACKGROUND

Figure 1 shows a schematic of a typical CVD process chamber. Process gases are fed into the chamber, where they react at a predetermined temperature and pressure to produce a coherent, uniform thin film on a substrate surface. Since all surfaces, including the chamber walls and internal chamber component surfaces are exposed to these gases and are at or near the deposition temperature, the chemistry that produces the thin film on the substrate also produces a thin film on these other

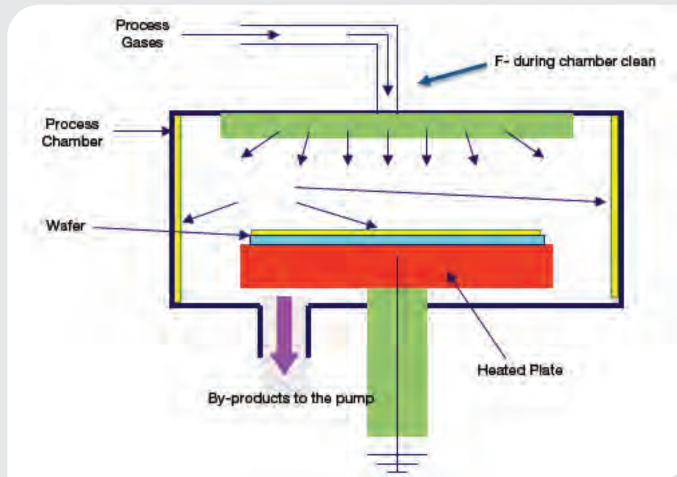

Figure 1 - CVD process chamber.

surfaces, the thickness of which grows over multiple process cycles. As the CVD chamber is cycled through different temperatures, interfacial stresses between the thick film and the component surfaces cause the film to fracture and delaminate, producing gas-phase particulates. These particles deposit on the substrate, producing defects that negatively impact the product yield.

Proper maintenance of CVD chambers requires material deposits on the chamber walls and components be periodically removed before they can delaminate and cause particle problems. This is accomplished using a periodic etch using reactive fluorine, oxygen, or other species produced in a plasma discharge within or attached to the process chamber. Reactive species remove solid deposits on the chamber walls and components by reacting with them to produce a gaseous product, e.g.:

$$Si^{+} + 4F^{-} \rightarrow SiF_4 \quad [1]$$

The optimum time required for a chamber cleaning process is a complex function of different variables including thickness of the wall deposits, temperature of the chamber surfaces, etch rate of the deposits at the temperature and pressure of the cleaning process, and the chemical composition of the material that is being removed. Ensuring that the cleaning process time matches only what's needed to remove all the material on the chamber surfaces is important. If the processing time leaves the chamber under-cleaned, material remains on the chamber surfaces that can accumulate and eventually produce particle contamination. If the processing time produces over-cleaning, the chamber surfaces can be etched producing metal fluoride particles that can contaminate subsequent CVD processes (Figure 2). Over-cleaning also wastes expensive reagent gases and etching of some components in the chamber (i.e., pressure and temperature sensors, mechanical wafer handling components, etc.) will shorten their lifespan leading to an increased operational cost of the CVD tool.

Chamber cleaning processes are affected by different parameters. The purity of the cleaning gas (NF_3, O_3, etc.) is important since impurities can both dilute the cleaning reagent and interfere with the cleaning chemistry. The physical parameters within the remote plasma reactive gas source determine the dissociation efficiency of the precursor gas and thus the concentration of the effective cleaning agent in the process chamber. The vacuum pumping characteristics and geometry of the process chamber are also important since these factors impact the recombination rate of the reactive species and the local chemical equilibrium of the etching reaction. Process variables, most notably temperature, can affect the cleaning process endpoint time by 5 – 10%. Additionally, some of these factors may vary over time which means that setting a fixed time for a chamber cleaning process doesn't work in a production environment.

The "sweet spot" between chamber under-clean and over-clean is quite short. Figure 3 shows a graphical depiction of chamber cleaning for a silicon-based CVD process. NF_3 is used as a source of reactive fluorine (via passage through a plasma) which etches the silicon deposits from the chamber walls and components according to Reaction [1]. The reaction byproduct, SiF_4, is monitored over time to determine when the cleaning process has been completed. In this case, the "sweet spot" is less than 10 seconds.

Consideration of the factors outlined above shows that ensuring the optimum chamber cleaning in the least amount of time requires an accurate, real-time endpoint sensor.

Chamber clean endpoint detection can be performed using different sensing methodologies. Optical Emission Spectroscopy (OES) has been successfully used for endpoint detection in cleaning processes that employ

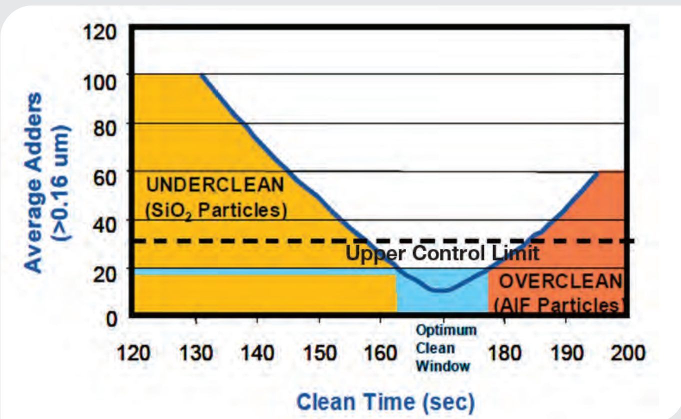

Figure 2 - The relationship between particulate contamination and chamber cleaning (AMAT HDP Ultima tool).

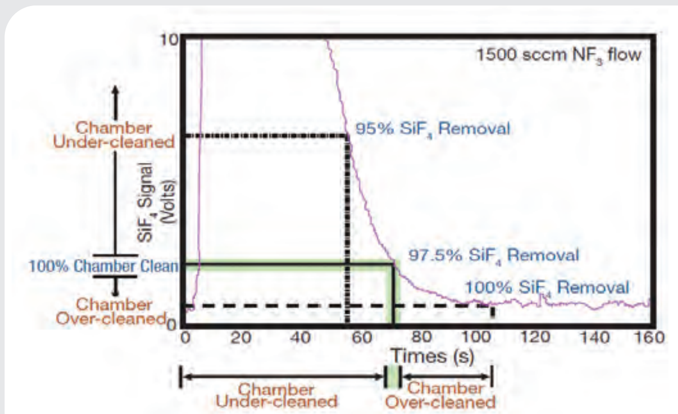

Figure 3 - A chamber cleaning cycle for the removal of silicon deposits using NF_3 and a remote plasma source.

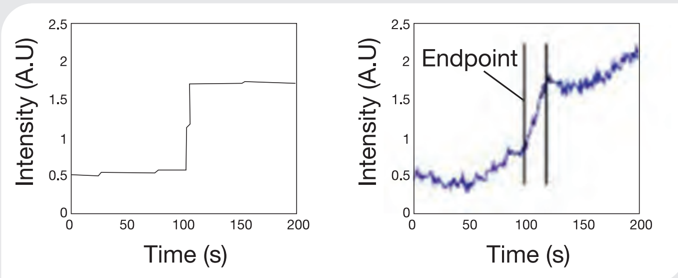

Figure 4 - Endpoint detection using OES.

in situ plasma excitation. It has also been used for endpoint detection in plasma etch processes. OES detects the light emitted when the electrons in gas-phase atoms or molecular fragments are first raised in energy by the absorption of energy from the plasma, then fall back to their normal energy level, emitting the previously absorbed energy as light. This light exhibits wavelengths and intensities that are characteristic of the atoms and molecular fragments present in the plasma. It can be recorded as a spectrum that can be analyzed to determine the chemical species in the plasma and their concentrations. By tracking the intensity of the optical signal due to the etching agent or etch byproduct in a chamber clean or plasma etch, OES spectrometry can determine the endpoint of the process (Figure 4). OES spectrometry for endpoint detection suffers from the relatively high optical noise level present in all plasma environments and is affected by plasma stability and uniformity.

Chamber clean and plasma etch processes can also use measurements of the plasma impedance as a means of endpoint detection. The detection mechanism in this instance depends on the fact that while gaseous F^- or other ions are being consumed in the cleaning/etching process, the plasma impedance remains relatively high. As soon as the ions are no longer being consumed in the clean/etch process, their concentration in the plasma increases, lowering the plasma impedance. Figure 5 shows a typical scan of plasma RF characteristics vs. time for an in situ plasma cleaning process with the endpoint indicated. Plasma impedance measurements can be impacted by many parameters in the chamber clean process (i.e., temperature, pressure, flow, etc.).

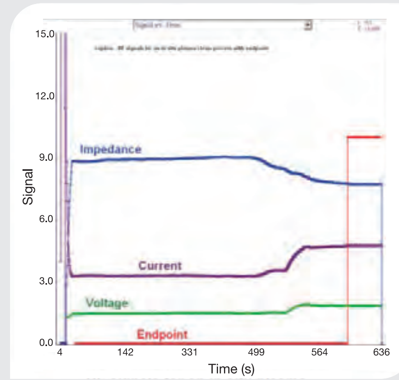

Figure 5 - RF signals for an in situ plasma cleaning process showing the process endpoint.

Finally, chamber clean endpoints can be detected using the characteristic infrared optical absorbance fingerprints of cleaning byproducts that are present in the exhaust gas stream. Figure 6 shows infrared absorbance measurements of the exhaust gases from F-based and O-based chamber cleaning processes. The scans

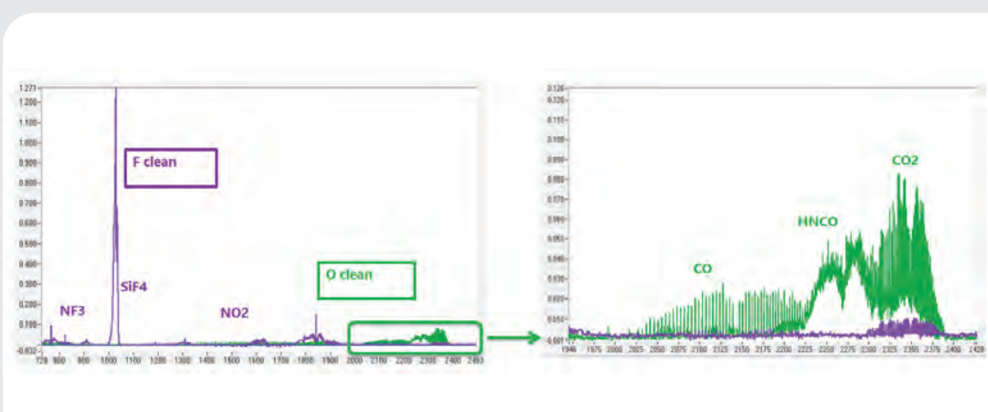

Figure 6 - Infrared absorption spectrum showing peaks that are present during F-based and O-based cleaning processes.

measure infrared absorbance against wavenumber, $^1/_\lambda$, where λ is the wavelength of the light in cm. The Full Scale scan on the left shows the strong Si-F IR absorbance due to the presence of the SiF_4 reaction byproduct in the exhaust stream from an F-based silicon CVD chamber clean. The expanded spectral region on the right shows the spectral signatures for similar products of an O-based chamber cleaning process. The intensity of the infrared absorbance is proportional to the concentration of the gas-phase species from which it arises according to the relationship:

$$\textit{Absorbance} = \log\left(\frac{I_0}{I}\right) \qquad (1)$$

where I_0 is the optical signal intensity without any absorption, and I is the optical signal intensity when the product of the cleaning process is present in the exhaust from the process chamber. Higher gas concentrations will cause a lower value for I and higher absorbance values. Thus, by analyzing the infrared light intensity variation at specific IR wavelengths, the concentration of a specific gas species can be precisely determined. When the monitored gas species is the byproduct of a chamber clean or etching process, the signal can be used for very precise endpoint detection. Figure 7 shows the basic components of an infrared absorbance-based system for endpoint detection. The chamber exhaust gas specimen is passed through a flow-through sample cell with a precisely known optical path length. The sample cell has IR-transparent optical windows on both ends. The infrared light source introduces the IR beam at one end of the cell, the beam passes through the sample gas in the cell, and then is detected by a spectrometer at the other end of the gas cell. The spectrometer separates the light by wavelength and measures the intensity at each wavelength.

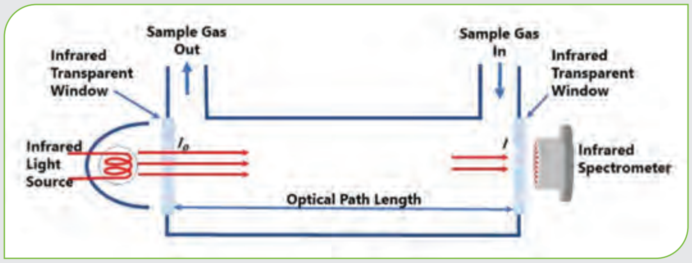

Figure 7 - Basic components of an infrared absorption spectrometer.

SOLUTION

MKS Instruments offers two chamber clean endpoint detectors based on infrared absorbance, the Process Sense™ Endpoint Detector and the T-Series Process Gas Analyzer (Figure 8).

Figure 8 - MKS Instruments' infrared-based chamber clean endpoint detectors.

The Process Sense Endpoint Detector is a small sensor specifically designed for chamber clean endpoint detection for CVD deposition chambers. It provides a simple analog output for the concentration of cleaning byproducts such as SiF_4 in the chamber exhaust flows with a sensitivity down to 1 ppm. The low detection limit makes the Process Sense well suited for chamber clean endpoint detection in silicon oxide (USG, FSG, PSG, BSG, BPSG), silicon nitride, polysilicon, and other silane- or TEOS-based CVD processes.

MKS NDIR technology (Figure 9) uses a broadband (blackbody) IR emitter and an optical filter that allows only in-band IR light to pass through (i.e., wavelengths specific to e.g., Si-F infrared absorption), measuring variations in the concentration of SiF_4 or another chamber clean byproduct (i.e. CO_2 in an O-based clean). Figure 10 shows a typical installation for a Process Sense Endpoint Detector. The sensor is mounted in an isolatable bypass arrangement. During the deposition

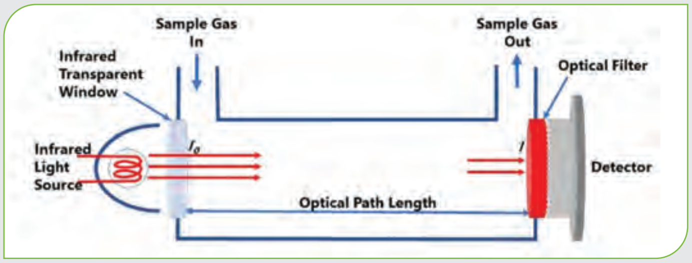

Figure 9 - A schematic of the gas sampling and analysis configuration used in the Process Sense™ NDIR endpoint detector.

process, the isolation valves are closed, protecting the sensor from contamination and coating by the deposition gases. During the chamber cleaning process, the isolation valves are open and the chamber clean byproduct gases flow through the detector's gas sampling cell. Once the absorption signal due to the chamber clean byproduct drops to zero, the chamber clean has been completed.

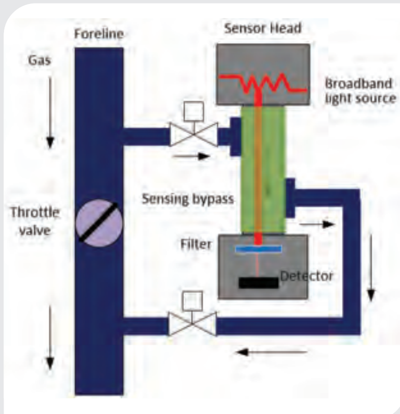

Figure 10 - Typical installation of the Process Sense™ Endpoint Detector.

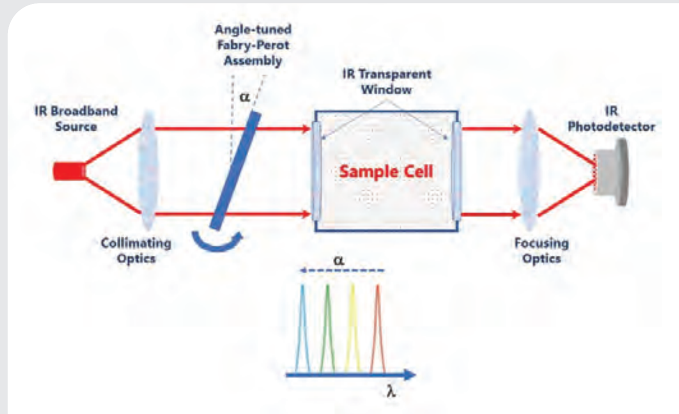

Figure 11 - Schematic of the optics configuration in MKS' T-Series Inline Gas Analyzer.

MKS Instruments' T-Series Inline Gas Analyzer (IGA) can be used for both inline process analysis and chamber clean endpoint detection. The T-Series IGA improves gas measurement accuracy over the traditional NDIR analyzers by using Tunable Filter Spectroscopy (TFS™), a spectroscopic scanning technique that can generate slices of spectra in selected regions of the infrared spectrum. TFS analyzers use a tunable Fabry-Perot Etalon assembly that enables high-throughput and high-precision wavelength scanning in one or more preselected band(s) of the infrared spectrum. The rotatable Fabry-Perot assembly can be adjusted through the angle α, shown in Figure 11, to allow different wavelength bands to be scanned. The TFS analyzer's wavelength separator is designed to produce an infrared probe that is tunable across a wavelength band of between 100 and 300 cm^{-1} (Figure 12). This allows the infrared probe to scan a portion of the infrared spectrum sufficiently wide to capture absorption data for individual group frequencies, while simultaneously minimizing interferences due to nearby absorptions. The T-Series IGA can be factory-configured for an α-value commensurate with real-time monitoring of the spectral regions corresponding to chamber clean byproducts

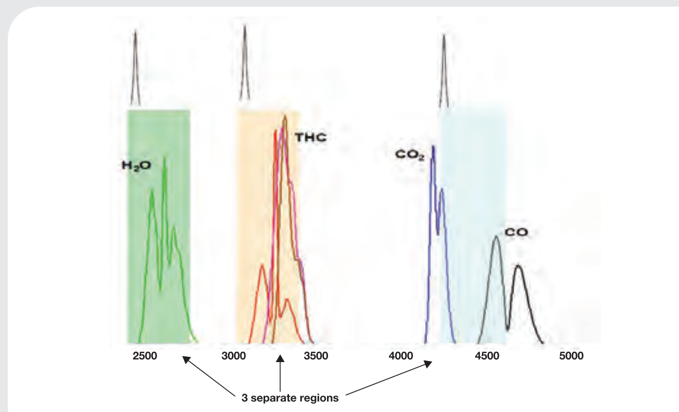

Figure 12 - TFS sensor wavelength scanning technology for the analysis of water, total hydrocarbon, CO_2, and CO concentrations in a gas sample.

such as SiF_4 or CO_2. These analyzers provide accurate and selective data for endpoint detection at sensitivities that can range between ppm and percent levels for most IR-active gases. T-Series Analyzers are supplied as complete integration-ready systems with inline or in-process sampling and configurable manifold and port designs that are easily installed and maintained.

Figure 13 shows typical T-Series IGA signal traces for O- and F-based chamber cleans. Before the cleaning process begins, the endpoint detector signal for the selected byproduct is at zero. Once the cleaning process is underway, the signal for the byproduct rises rapidly and remains high until the cleaning process is complete, at which point it drops back to zero or near zero.

Chamber clean byproduct profiles are highly repeatable with no dependence on the plasma or process conditions in the chamber cleaning process.

Table 1 provides a summary of the detection sensitivities of Process Sense NDIR Endpoint Detector and the T-Series Inline Gas Analyzer for chamber clean endpoint analysis. Table 2 provides guidance for the best choice of MKS Instruments' IR endpoint detection tools in different applications.

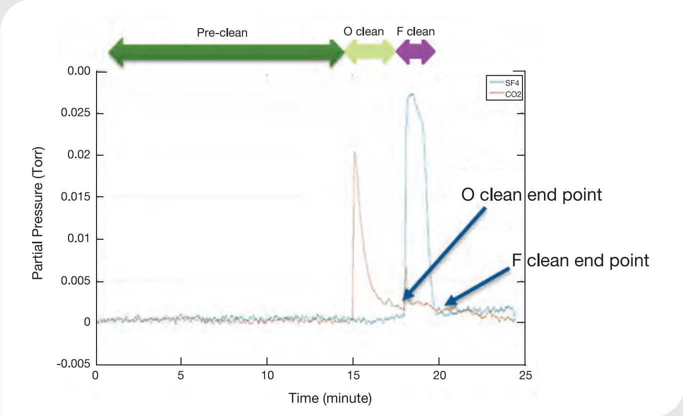

Figure 13 - Typical T-Series IGA signal traces for O- and F-based chamber clean.

Filter Type	Application Example	Gases	Process Sense Capability (mTorr)	T-Series Inline Gas Analyzer Capability (mTorr)
Single	Dielectric	SiF_4	1.5	0.4
Single	Hardmask	CO_2	N/A	0.4
Single	WCVD	WF_6	2	0.8
Dual	Low K	$SiF_4 + CO_2$	N/A	SiF_4 : 0.4 CO_2 : 0.6
Single	Mo CVD	MoF_6	In Development	In Development

Table 1 - IR EPD's sensitivity for different gases (based on 1 second and 5 second integral time).

Applications	MKS Solution
SiO_2/SiN Dielectric Deposition Chamber Clean	Process Sense/IGA-T-SiF_4
Amorphous Carbon Layer Deposition Chamber Clean/ Hardmask Etching	IGA-T-CO_2
W Deposition Chamber Clean	Process Sense/ IGA-T-WF_6
Low K Application – SiCN Deposition Chamber Clean	Process Sense/Dual band IGA-T-SiF_4-CO_2

Table 2 - MKS Instruments' solutions for chamber clean endpoint detection applications.

CONCLUSION

Efficient and complete chamber cleaning processes are critical for successful ALD/CVD processes. These processes have a very short temporal "sweet spot" for process shut-down to avoid problems such as high particle counts due to over-cleans. This means that an accurate, real-time endpoint detector is needed that provides a reliable, accurate, and process-independent indication of when the cleaning process has completed. Endpoint detectors that are based on infrared spectroscopy have advantages in that they offer high accuracy and repeatability, and their measurements are not affected by variations in process and plasma conditions. MKS Instruments' infrared-based endpoint detection tools, the Process Sense NDIR Endpoint Detector and the T-Series Inline Gas Analyzer are ideally suited for real-time monitoring of process chamber exhaust streams for chamber clean byproduct gases that can be used as endpoint indicators. These endpoint detectors have detection limits that vary between ppm and percentage levels. They are easily installed and maintained in configurable exhaust gas bypass systems that result in low cost of ownership for these endpoint detection systems.

APP NOTE - Chamber Clean Endpoint Detection_07/22