Multiple Gas Impurity Analyses and Verification For Semiconductor Process Tools

BY RICHARD T. MEYER

With the increased emphasis on the purity of cleaning, etching and treatment gases by the semiconductor industry, advanced analysis instrumentation is required to monitor the electronic specialty gases and to verify the performance of process tools and components. This paper describes new instrumentation and software specifically designed to accomplish ppb level gas analyses in times as short as 15 seconds.

The overall system description is called IRGAS™, which stands for "Integrated Real-Time Gas Analysis Solution." The IRGAS is a turnkey industrial gas analysis system. It is composed of a FTIR spectrometer, a stainless steel long path gas cell, optics, sample and purge gas lines and manifold, and two software components known as SPGAS™ and SpectraStream™. The SPGAS software provides two major functions: (1) total operational management and monitoring of all the hardware components; and (2)

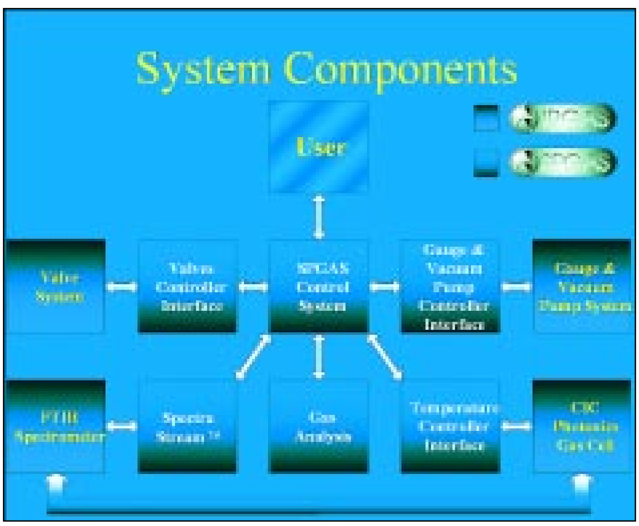

Fig. 1

weighted, multiband, multicomponent chemometrics-based quantitative analysis. The SpectraStream module adds the capability for a fast-response early-warning detection of sudden changes in gas purity or composition. A block diagram is shown in Figure 1.

The hardware components are well-established tools. The FTIR spectrometer is a Bomem WorkIR, which is a very compact and ruggedized unit frequently used in industrial applications; and the long path gas cell is a

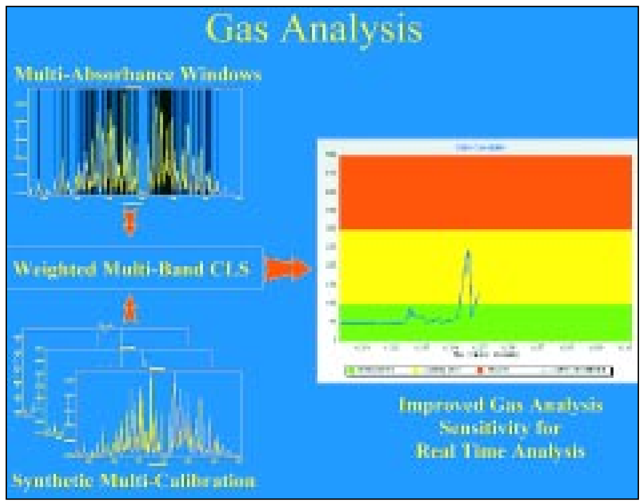

Fig. 2

stainless steel cell with customized gold-coated stainless steel mirrors enhanced to resist corrosive and toxic gases, to provide for fast exchanges of gas samples, and to offer very high energy throughput.

The SPGAS software offers gas calibrations based upon fundamental HITRAN data for up to 36 gaseous species, weighted multiband CLS (classical least squares) data analysis, protection against unknown species, and infinite calibration sets. It automatically matches, in real time, the recorded sample gas spectra with calibration spectra and then displays the absolute concentrations as a function of time, as illustrated in Figure 2. Multiple species can be simultaneously displayed. Two constraints of SPGAS are that the HITRAN data base is limited to those gaseous species found in atmospheric air and that the calibration sets must be regenerated for optimal performance if either the spectrometer or gas cell is changed; however, additional gas calibrations can be incorporated using standard calibration procedures.

The major benefits of the SpectraStream module are (1)

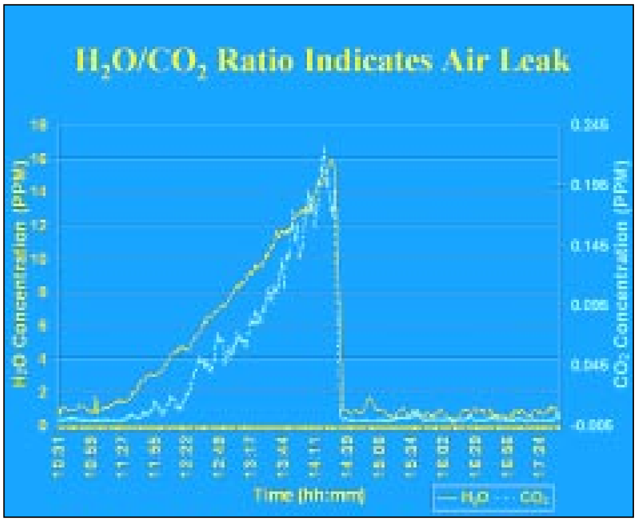

Fig. 3

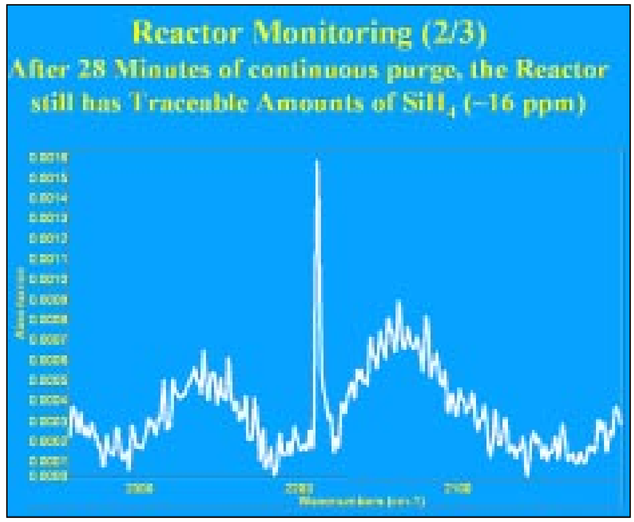

Fig. 5

that it provides high sensitivity detection of impurities at the low ppb level, (2) that it reduces the time response typically associated with FTIR spectroscopy from minutes to seconds, and (3) it reduces the effects of spectrometer drift.

Among other tests, SpectraStream has been applied to the rapid detection (</= 20 sec) of gas surges due to cylinder tank openings, including moisture surges from UHP nitrogen, and more importantly to the detection of air leaks in process gas lines. While FTIR can not detect O_2 from an air leak, it can detect both H_2O and CO_2 and use the rates of signal increase and the absolute ratio of the two species to prove the occurrence of an air leak. An actual result from a fab plant operation is shown in Figure 3. In this case, the increase in the CO_2 concentration (right scale) is the most obvious indicator of a leak. Then the identical growth rate pattern for H_2O is the concurrent indicator. But the proof of an air leak is given by the H_2\O to CO_2 ratio, which corresponds approximately to the air ratio at the plant site.

> **"The detection of an unexpected impurity in the reactor is another example of the power of the online gas analysis system."**

The greatest value of the IRGAS/SPGAS/SpectraStream system to the semiconductor industry is in its direct application to the performance of

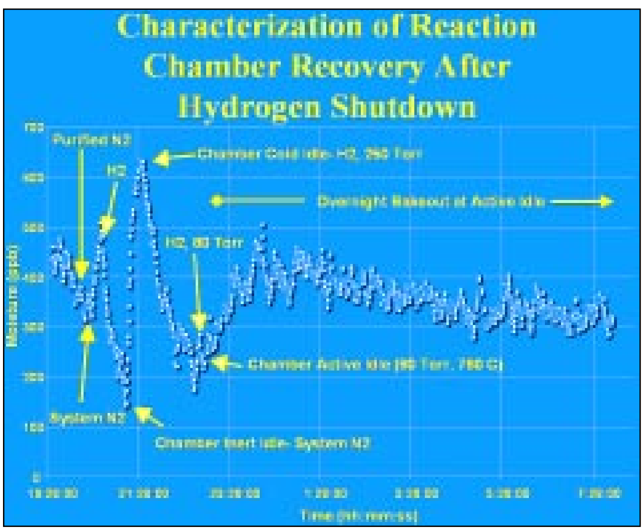

Fig. 4

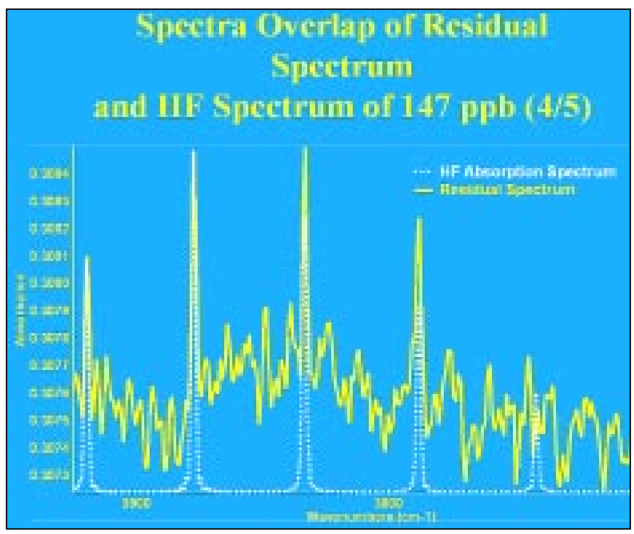

Fig. 6

process tools. The following data were collected from an epitaxial reaction chamber supplied with both system and purified nitrogen for purging, purified system hydrogen as a carrier gas, and various epitaxial treatment gases, including silane, dichlorosilane, germane, etc.

Figure 4 illustrates the characterization of the reaction chamber recovery after an unscheduled hydrogen shutdown. It records the moisture concentrations in ppb within the reactor as various cycles of system N_2, purified N_2, and system H_2 are activated and as the reactor temperature and pressure are adjusted. The associated time scale here is hours, which is the real time scale over which the epitaxial reactor was reconditioned for use.

Figure 5 reveals that hydrogen purging of the reactor for 28 minutes after a treatment with SiH_4 (200 sccm in 60 slpm H_2) does not clear the reactor of all SiH_4, as was held to be the case before these measurements. Traceable amounts of SiH_4 of approximately 16 ppm were still present.

The detection of an unexpected impurity in the reactor is another example of the power of the online gas analysis system. Figure 6 is one of several SPGAS records used to isolate four residual absorption peaks within the spectral region of moisture absorption. The HITRAN data base was used to identify these peaks as due to HF at a concentration of about 147 ppb. However, no HF had ever been used with this reactor. The ultimate conclusion was that the elastomer seals in the reactor tool were being degraded, with HF being one of the chemical degradation products.

Moisture as an impurity in process gases remains one of the greatest concerns in the semiconductor industry. In most applications today, moisture levels of 100 ppb or lower are sought for the treatment gases. Figure 7 records the moisture content of several carrier and treatment gases as they are cycled through an epitaxial reactor. One can readily observe that the system H_2 (carrier) contains moisture at levels below 50 ppb and reaching 20 ppb in certain cycles. However, the moisture levels in HCl, SiH_4, DCS, and GeH_4 exceed 100 ppb, reaching 150 ppb in some instances. Once again the capability of the real-time, online IRGAS System is demonstrated.

Since it is important to verify that the IRGAS system itself is performing properly, a number of system validation parameters are available for monitoring. These include: IR light beam intensity; background moisture in the FTIR and gas cell; noise levels due to vibration and electronics; for monitoring the gas cell—energy throughput, mirror degradation, and window or mirror contamination; and lastly, the variation in the standard errors associated with the estimation of concentrations.

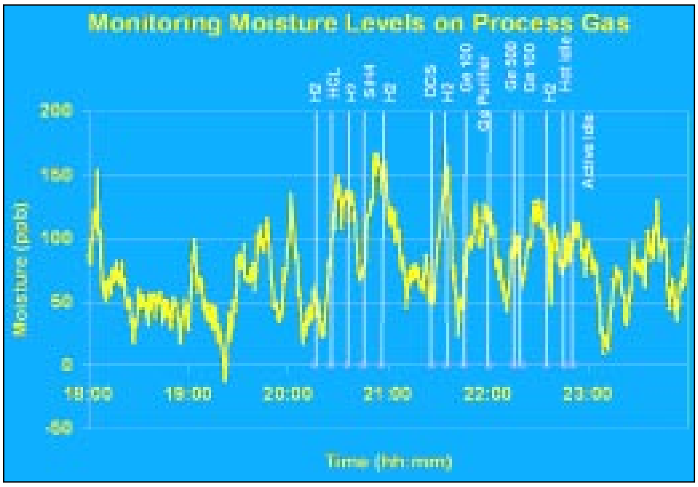

Fig. 7

In summary, the real values of the IRGAS system are: (1) that it permits simultaneous detection of all infrared-active gaseous species including process gases and their impurities, (2) that the use of SpectraStream provides very fast response time necessary for process monitoring, and (3) that the system is applicable to all corrosive and toxic gases, as well as other vapors. Indeed, its application as a foreign agent chemical detection device is also being developed and tested.

Richard T. Meyer, Ph.D., is CEO and President of CIC Photonics, Inc., 3825 Osuna Rd NE, Suite 6, Albuquerque, NM 87109 . He can be reached at 505-343-9500 or sales@cicp.com, www.irgas.com.