SRS Tech Note

A comparison between Stanford Research Systems' BGA244 Binary Gas Analyzer and Inficon's Composer Elite Gas Concentration Monitor. (Compiled by Stanford Research Systems)

Overview

The speed of sound can be used to accurately determine the composition of binary gas mixtures. The technique has been used to measure the concentrations of MOCVD (metalorganic chemical vapor deposition) precursors in carrier gas flows for the semiconductor industry. This Note will compare two systems: Inficon's Composer Elite Gas Concentration Monitor and SRS's BGA244 Binary Gas Analyzer.

Inficon's Product

The Inficon Composer Elite Gas Concentration Monitor targets the measurement of dopant precursors such as trimethyl indium (TMIn) in semiconductor applications. The instrument determines the precursor concentration in a carrier gas by measuring the resonant frequency of a Helmholtz resonator through which the gas flows. The resonant frequency is proportional to the speed of sound in the gas, which is reduced by the presence of a heavy precursor in the gas stream.

SRS's Product

Stanford Research Systems' BGA244 Binary Gas Analyzer is intended for a broad range of applications. The instrument determines the speed of sound by measuring the resonant frequency of a gas filled cavity. Thermodynamic data for about 500 gases and liquids allows the analysis of tens of thousands of mixtures. The instrument's sensitivity, stability, accuracy and metal seals allow it to address many applications in semiconductor process control including the measurement of dopant precursors in carrier gases.

Speed of sound in an ideal gas

The speed of sound in an ideal gas is given by Equation 1:

$$W = \sqrt{\frac{\gamma\, R\, T}{M}} \qquad \text{Equation 1}$$

Where W is the speed of sound (m/s), γ the ratio of heat capacities ($\gamma = C_p/C_v$), M the molar mass (kg/mol), T the absolute temperature (K), and R the ideal gas constant (8.3144621 J/K-mol). The γ and M for the mixture are determined by the properties of each gas and their mole fractions within the mixture. By measuring the speed of sound and the temperature of a binary gas mixture, and knowing the properties of both gases, the mole fractions of the constituents can be precisely determined.

The speed of sound in an ideal gas is independent of pressure. This is not the case with real gases. Intermolecular forces create pressure dependencies to the speed of sound in real gases, an effect which is accounted for in the BGA244's gas composition analysis (but not accounted for by the Inficon product).

Pre-cursor sensitivity

MOCVD Pre-cursor materials have lower $\gamma's$ and much higher masses than their carrier gases (typically nitrogen or hydrogen). Both factors reduce the speed of sound. Composition measurements depend on the difference in mass of the pre-cursor and carrier gas and on the sensitivity, stability, and accuracy of the sensor's frequency and temperature measurements.

This Note will examine many factor which effect the measurements of MOCVD precursors in carrier gases. The examination will show that the SRS instrument has a tenfold advantage in sensitivity, stability, and accuracy for both temperature and frequency measurements relative to the Inficon instrument. Further, the SRS instrument is unaffected by atmospheric pressure changes and is largely immune to changes in process pressures as well.

Specification comparison

The spreadsheet on the next page compares the important features and characteristics of the two instruments. A detailed discussion will follow.

Comparison between SRS BGA244 Binary Gas Analyzer and Inficon Composer Elite Gas Concentration Monitor

Specification	SRS BGA244	Inficon Composer Elite
Pressure and Flow		
Recommended operating pressure (N2)	3.6 to 150 psia	3.8 to 19.3 psia
Routine over-pressure	1,000 psia	29.4 psia
Damage pressure	> 2,500 psia	> 120 psia
Allowed flow rate (helium or hydrogen)	0 to 5,000 sccm	0 to 2,000 sccm
Allowed flow rate (nitrogen or heavier)	0 to 5,000 sccm	1,000 to 2,000 sccm
Internal volume	130 cc	12.6 cc
Frequency measurement		
Frequency stability	± 5 ppm	± 45 ppm
Frequency error from transducer artifacts	0.1% (10 to 150 psia)	3.8% (10 to 20 psia)
Measurement method	FFT with fitting	Stepped sine with fitting
Measurement rate	4.39 Hz	1 Hz
Measurement averaging	1 to 100 samples	1 to 100 samples
Gas composition analysis		
Tabulated gases and liquids	500	36 (Listed in manual)
Binary gas composition analysis	Yes	As a precursor monitor only
Specific heat temperature corrections	Yes	No
Acoustic virial corrections	Yes	No
Transducer resonance corrections	Yes	No
Thermo-viscous boundary corrections	Yes	No
Temperature		
Ambient operating temperature	-20 C to + 70 C	+20 C to + 50 C
Max cell heater set point	70 C	65 C
Gas temperature measurement/stability	± 0.001 C	± 0.01 C
Mechanical and Power		
Gas fittings	VCR-4, VCO, NPT, or swage	VCR-4
Low power operation	USB power only (+5V, 320 mA)	Not available
Full power operation (with heaters)	+24 V / 2.5 A	+24 V / 6.25 A (for 5 sensors)
Sensor	Single unit, 80 cu-in	Cabled to controller, 130 cu-in
Controller	None required	Rack-mounted, 3U, half width
Insertion length (VCR fittings)	4.75"	4.88"
Sensor weight	7 lbs.	5 lbs.

Pressure and flow rates

The Inficon instrument uses thin (0.00067") Inconel diaphragms to separate the process gas from its acoustic transducers. These diaphragms assure that the process gas only contacts stainless steel or Inconel, but the diaphragms introduce some problems and limitations: The instrument can be damaged at pressures above 120 psia, the recommended operating pressure range is limited to 19.3 psia, and the diaphragms cause significant (3.8 %) pressure dependent frequency errors and create a sensitivity to atmospheric pressure. (This 3.8 % frequency error is huge when compared to Inficon's stated 0.0045 % stability specification.)

The SRS instrument uses acoustic transducers formed from 0.001" thick Kapton film with ENIG plated traces placed between nickel plated NdFeB magnets inside of a 1 cm thick stainless steel cylindrical gas chamber. The chamber has a proof pressure above 2,500 psi. Since the transducers are inside the chamber they are undamaged by over-pressure conditions or rapid de-pressurization.

The SRS instrument has been tested for flow rates from 0 to 5000 sccm and can be used for light or heavy gases (from H_2 to SF_6). Inficon sells two versions of their gas sensor. The model for hydrogen and helium can be used between 0 and 2000 sccm while the heavy gas model (N_2 and above) requires a minimum flow rate of 1000 sccm.

The SRS instrument has a larger gas volume, 130 cc vs. the Inficon's instrument with 12.6 cc. Larger gas volumes will slow response times at low flow rates. However, the larger volume allows a cavity with a much higher Q-factor, which allows a more accurate determination of the resonate frequency (hence speed of sound). This larger cavity is one of the factors which allows the SRS instrument to have a tenfold better stability when compared to the Inficon instrument.

Temperature measurement and control

Both the speed of sound and the temperature must be measured in order to determine the gas composition. The two instruments take different approaches to the measurement (or control) of the gas temperature.

The Inficon instrument uses two controlled heaters (one on the inlet tube, the other in the resonant chamber) to control the temperature of the gas during the measurement. The heaters must always be used with their set-points above ambient. Careful adjustment of set-points is required to minimize the change in resonant frequency vs. flow rate.

The SRS has heaters also, but they are not required to be used to make an accurate measurement. (They used to accelerate vacuum outgassing and to prevent condensation from the process gas inside the resonance cavity.) To measure the gas temperature, two glass-beaded thermistors are suspended in the gas at opposite ends of the resonance cavity. The thermistors are calibrated to the speed of sound in argon over the ambient operating temperatures (-20C to +70C). The high sensitivity of the thermistors provide millidegree temperature resolution and fast response times. Measuring the gas temperature (instead of trying to control it) reduces the error associated with a changing flow rate.

Frequency measurements

The two instruments take very different approaches to the frequency measurements. SRS uses a DSP based Fast Fourier Transform (FFT) network analyzer to measure the entire acoustic spectrum, from DC to 36,000 Hz, simultaneously. A real-time correlation analysis is done to identify the cavity resonances. This allows the instrument to track changes automatically without the need to switch modes. Lorentzian profiles are fit to the spectral data providing a ±5 ppm frequency stability.

The Inficon uses a swept frequency measurement around the Helmholtz resonance to determine the resonance frequency with a ±45 ppm stability. This stability is nine-times worse than the SRS instrument. The instrument cannot make continuous measurements if the gas composition is changing quickly, rather, it enters into a "QTrack" mode (with decreased accuracy) or the "SEARCH" mode (which provides no useful measurement data).

Pressure sensitivity of transducer artifacts

Both instruments exhibit a frequency measurement error vs. operating pressure caused by resonances in their acoustic transducers. The Inconel diaphragms used in the Inficon instrument cause a peak-to-peak frequency deviation of about 150 Hz, or about 3.8 %, over the operating pressure range of 200 – 1000 Torr as shown in Figure 1.

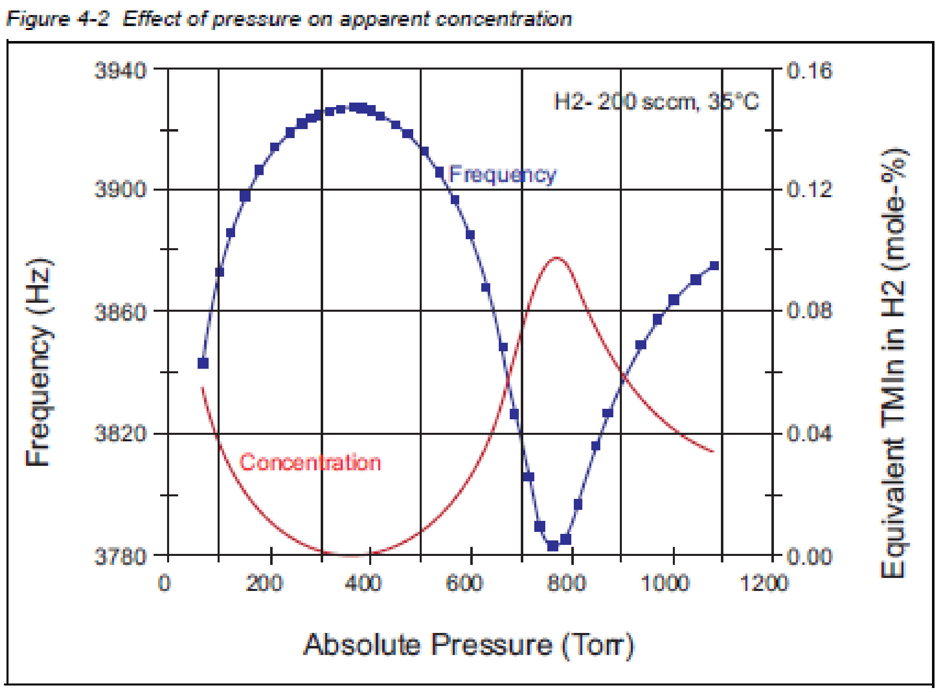

Figure 1. Inficon's pressure induced errors (about 3.8%).

This error is not corrected in the Inficon instrument and so can cause an equivalent concentration error of up to 0.10 % mole equivalent of TMIn over the operating pressure range (as shown by the red trace Figure 1). This error dwarfs the stated "reproducibility" of 0.0003 % or "sensitivity" of 0.0001 % and so small changes in pressure (or atmospheric pressure) will require the instrument to be "re-rel'd" to avoid erroneous results.

SRS Transducer artifacts and corrections

The acoustic transducers used in the SRS instrument cause a much smaller frequency shift as shown in Figure 2. There are three pressure tuned artifacts: A transducer resonance (TR%), an reduced mass correction (RM%), and a thermos-viscous (TV%) shift. Uncorrected, the three effects sum to less than 0.50 % over the operating pressure range of 150 psia. Compared to the Inficon device, this is about a 7x smaller pulling error and a 7x larger operating pressure range.

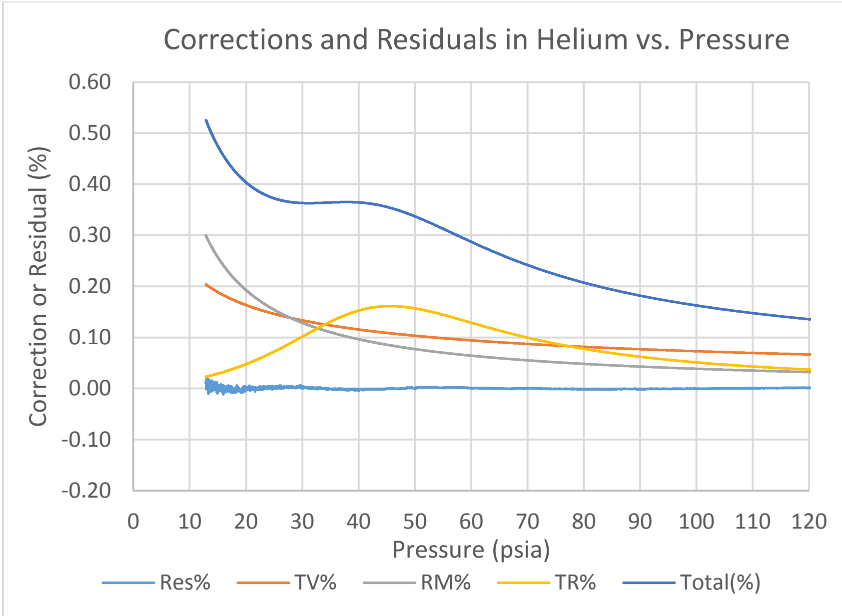

Figure 2. SRS frequency corrections vs pressure. The residual error is less than ±0.02%

Additionally, the BGA244 automatically compensates for these effects (when connected to a pressure gage), leaving a residual error of less than ±0.02% (Res%) which is about 190 times smaller than the Composer Elite's error. An example of these automatic corrections at work is shown in Figure 3. The graph shows the measured (uncorrected) speed of sound in helium vs. pressure together with the corrected speed of sound and the NIST REFPROP value (which lie directly on top of one another).

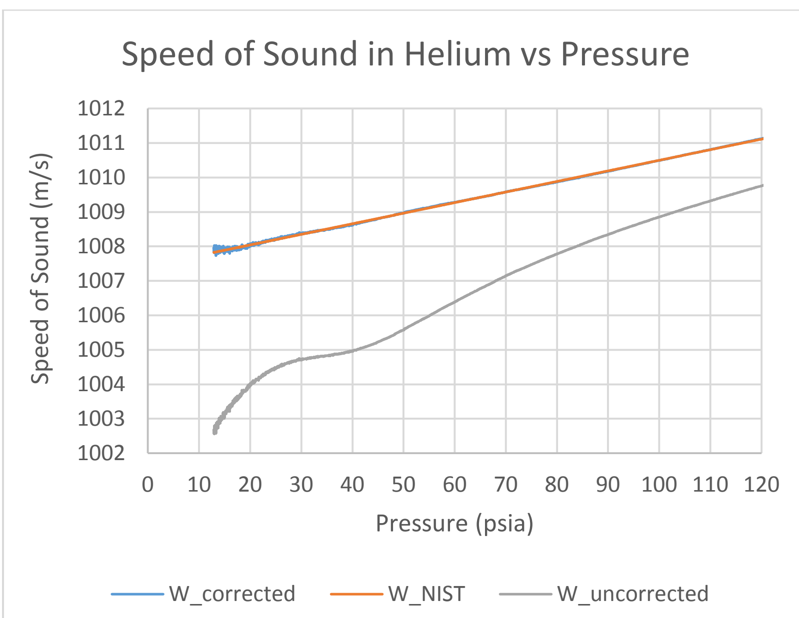

Figure 3. Speed of sound in helium vs pressure.

Gas composition analysis

The SRS instrument was designed to make accurate gas composition measurements knowing only which gas species are present. For example, the user can specify that the mixture is composed of CH_4 and N_2, and by measuring the gas temperature and the speed of sound (and knowing the pressure) the instrument will be able to measure the composition to better than 0.1%. To do this, the instrument uses thermodynamic data for the gases (data for more than 500 gases are in the instrument's memory). For accurate analysis more than γ and the molar masses are needed: To calculate an accurate composition the instrument also uses the temperature dependence of heat capacities, acoustic virial corrections (the speed of sound in real gases changes with pressure as shown in the speed of sound in helium vs pressure graph above), corrects for thermo-viscous boundary layers and for the frequency pulling by transducer resonances. All of these effects are accounted for in the SRS instrument and none of them are accounted for in the Inficon instrument.

Relative measurements

The BGA244 will provide "out of the box" binary gas composition accuracy of about ±0.02 % for TMIn in hydrogen. To eliminate this source of error the instrument has a "Rel" mode: The user flows the pure carrier gas and places the instrument into the "Rel Mode". The instrument uses the change in the speed of sound from the Rel'd value to compute the concentration of the TMIn in all future measurements. The instrument will not need to be re-zeroed.

The Inficon product also supports a Rel'd Mode. Depending on accuracy requirements, re-zeroing may be required frequently to offset errors caused by daily atmospheric pressure changes.

Form factor and power

The SRS instrument integrates the gas sensor and controller into a single 3.25" x 4.5" x 5.5" inch device. The unit is available with or without a touch-screen graphics display as shown in Figure 4. All functions and measurements are available via RS-422, RS-232 or USB interfaces. The unit can be powered from a single USB port (without heaters) or from a +24 V power supply (which provides heater power and enables analog interfaces.)

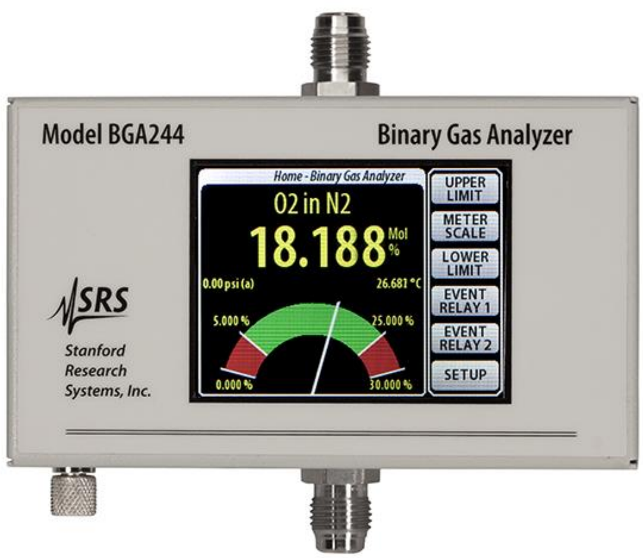

Figure 4. BGA244 Binary Gas Analyzer, top view, showing touch screen GUI.

The Inficon device consists of two units: A 3.06” x 3.29” x 6.89” gas sensor module and a 3U half-rack control unit connected with a 15’ cable. Each rack-mounted control unit can operate up to five gas sensors. The rack-mounted control unit can use either RS-232 or DeviceNet (when an optional adapter card is installed).

System integration

The SRS BGA244 has several optional features which can assist with system integration as shown in Figure 5. There are two analog inputs and three analog outputs, and two user defined event relays for process control or alarms. The analog inputs and outputs have 0-5 V, 0-10 V and 4-20 mA ranges. Inputs have adjustable loop power supplies and may be used to read pressure gauges, other sensors, or the outputs from other instruments. Outputs may be used to transmit measurements or to control flow valves, etc.

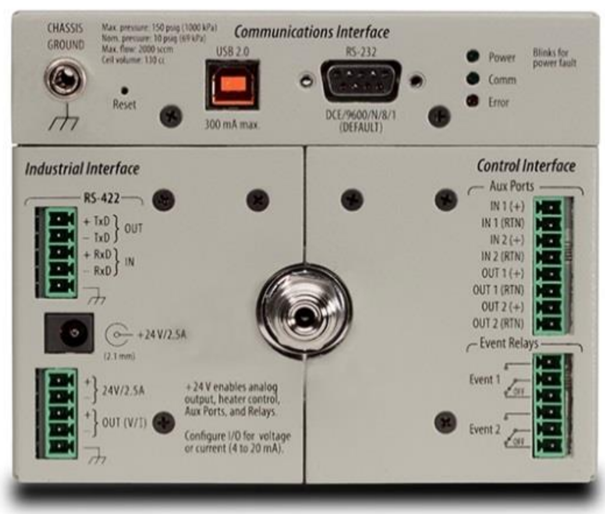

Figure 5. Standard USB and RS232 interfaces and optional RS422, 4-20 mA and +24 v power

The analog inputs allow the instrument to measure process gas pressure with a 3rd party pressure gage. The pressure can be displayed on the BGA244, read via the USB, RS232, RS422, or transmitted on one of the 1-10 V or 4-20 mA analog output. The pressure reading is also used to make speed of sound corrections (for transducer, thermoviscous and virial effects) improving the accuracy of binary gas compositional analysis.

Appendix: Converting frequency stability to mole fraction stability

The above discussion focused on frequency stability of the two instruments. Users care about the stability of the inferred mole fraction of the metal-organic precursors in the carrier gas. Here's how to convert between the two…

The measured frequency is proportional to the speed of sound in the cavity:

$$f \propto W = \sqrt{\frac{\gamma\, R\, T}{M}}$$

For the purposes of stability analysis, when a small amount of a heavy pre-cursor is added to a lighter carrier gas the frequency shift is dominated by the change in mass (we'll ignore the change in γ here). With that,

$$\frac{dW}{dM} = \frac{-1}{2M}\sqrt{\frac{\gamma\, R\, T}{M}} = \frac{-W}{2M} \quad and \quad \frac{df}{dM} = \frac{-f}{2M}$$

For a mixture of components $1 + 2$, the molar mass of the mixture is given by:

$$M_m = M_1 x_1 + M_2 x_2 = \ M_1 x_1 + M_2(1 - x_1)$$

Let x_1 be the (small) fraction of the pre-cursor in the carrier gas, M_1 its molar mass, and M_2 the molar mass of the carrier gas. Then, the change in the molar mass of the mixture is given by:

$$\Delta M_m = \ M_1 x_1 + M_2(1 - x_1) - M_2 = \ (M_1 - M_2) \cdot x_1$$

Then, the change in frequency caused by adding the pre-cursor to the carrier is given by:

$$\Delta f = \frac{df}{dM} \cdot \Delta M = \frac{-f}{2M} \cdot \Delta M \approx \frac{-f}{2M_2} \cdot \ (M_1 - M_2) \cdot x_1$$

Or

$$x_1 = \ -\frac{\Delta f}{f}\frac{2M_2}{(M_1 - M_2)} = -0.424 \cdot \frac{\Delta f}{f}\ (for\ TMIn\ in\ N_2) = -0.0253 \cdot \frac{\Delta f}{f}\ (for\ TMIn\ in\ H_2)$$

The final equation shows that the mole fraction of the pre-cursor is proportional the fractional frequency shift, $-\Delta f/f$ with a scale factor of $2M_2/\ (M_1 - M_2)$. The scale factor is 0.0253 for TMIn in H_2 and 0.424 for TMIn in N_2.

Sensitivity, reproducibility and stability

For TMIn in N_2, Inficon operation manual states a "composition reproducibility" of 24 ppm and a "sensitivity" of 8 ppm. The product brochure states a frequency stability of 45 ppm which, using the factor of 0.424 derived above, would indicate a "mole fraction stability" of 19 ppm. The values for "composition reproducibility" and "mole fraction stability" (24 ppm and 19 ppm) are reasonably consistent, however these errors are dwarfed by errors caused by operating or atmospheric pressure changes.

The SRS BGA244, with its measured frequency stability of ± 5 ppm compared to Inficon's ± 45 ppm, promises a substantially better mole fraction stability. Further, the BGA244's temperature and pressure correction algorithms offer a real-world "composition reproducibility" advantage by removing virtually all pressure dependencies.