OPTIX Robust & Easy to Use Residual Gas Analysis for the Vacuum Industry

• ALD
• Atmospheric vacuum sampling via roughing
• Contamination check
• CVD
• Etch Endpoint
• Heat Treatment
• Leak Detection (any species)
• Plasma Treatment
• Process Gas Analysis
• OLED
• MOCVD
• PVD
• Plasma Spraying
23 Years of Products and Technology from Gencoa

Rotatable & Planar Magnetron Sputter Cathodes • Retrofit magnetic packs • Plasma Treaters • Speedflo Reactive Gas Controllers • IM Ion Sources & power supplies • Arc MAX sources & power supplies • Active Anodes and Gas Delivery Bars • OPTIX Gas and Chemical Sensing • S and Se Sensor • PEC Pulsed Effusion Cell • V’DLC - Transparent DLC • IC Nano antimicrobial layer technology • Process implementation & tuning •

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Why Monitor Your Vacuum Process?

Save Money by Avoiding Problems

- Identify vacuum or process problems before they have a financial impact
- OPTIX maps the process environment to ensure reliable production
- Improves quality of products and repeatability
- Outputs for better process control
OPTIX vs RGA

OPTIX – remote plasma gas analysis (RPGA)
Optical method - high speed
Detector outside of the vacuum – cannot be damaged by the vacuum environment

Quadrupole Residual Gas Analyzers (RGAs)
Detector inside of the vacuum – will be damaged by the vacuum environment by high pressure, operator error, or contamination

Remote plasma

Low ppm detection

Vacuum window

Light

Spectrometer

Low ppm detection
# OPTIX vs RGA

<table>
<thead>
<tr>
<th>OPTIX – remote plasma gas analysis (RPGA) Optical method</th>
<th>Quadrupole Residual Gas Analyzers (RGAs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Robust— detector separated from chemicals by optical window</td>
<td>Detector in contact with chemicals – easy to contaminate, hard to clean</td>
</tr>
<tr>
<td>No filaments – simple electrode geometry</td>
<td>Filaments and ionizers are consumables</td>
</tr>
<tr>
<td>Operates 0.5 to 10⁻⁶ mbar</td>
<td>Only operates reliably down to 10⁻⁴ mbar</td>
</tr>
<tr>
<td>Direct chamber monitoring – no need for differential pumping unless atmospheric sampling</td>
<td>Higher than 10⁻⁴ mbar pressure needs differential pumping – loss of sensitivity</td>
</tr>
<tr>
<td>FAST – ‘speed of light’, 10-50 msec response</td>
<td>Typically 0.5 to several seconds range</td>
</tr>
<tr>
<td>Tolerates volatiles in the vacuum – hydrocarbons, solvents, long chain polymers</td>
<td>Only small amounts of contamination before sensor failure</td>
</tr>
<tr>
<td>Wide range of useful software applications available – gas tracking, leak detection, pump-down monitoring, water tracker, end-point detection, multi-mode process tracking</td>
<td>Typically gas tracking &amp; leak detection</td>
</tr>
<tr>
<td>Sensor degassing mode – avoid false reading</td>
<td>Yes, but degas can affect filament lifetime</td>
</tr>
</tbody>
</table>
Plasma has some non-linearity.

Plasma light intensity too low

1E-6 mbar – 0.5 mbar

Vacuum process chamber 0.5 to 10^{-6} mbar or with a rotary pump to support atmospheric sensing

Wide pressure range remote plasma generator

High intensity plasma

Wide range spectrometer 200-850nm

Spectrum analysis gives species composition

OPTIX Remote Plasma Gas Analysis

RPGA

Spectrum analysis gives species composition
OPTIX leak detection and new applications outside conventional RGA technology

Unlike RGA’s the OPTIX detector is separated from the plasma and chemical environment by a physical window. Hence the detector is not affected by the nature of the media to be analyzed. As long as the plasma can be sustained & light is visible, chemical information can be obtained. This results in a much more rugged device.

<table>
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<tr>
<th>Leak checking</th>
<th>Volatile deposition processes</th>
<th>End-point</th>
<th>Chemical analysis from atmosphere</th>
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<tbody>
<tr>
<td>Other gases such as N₂</td>
<td>OLED, CVD</td>
<td>Etching</td>
<td>Gas chromatography</td>
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<tr>
<td>Refrigerant and air conditioning systems</td>
<td>ALD, MOCVD</td>
<td>Process gas consumption</td>
<td>Liquid and fuels composition analysis</td>
</tr>
<tr>
<td>Fuels and oil leaks from components</td>
<td>Flash evaporation</td>
<td>Freeze drying</td>
<td>APCI Atmospheric pressure chemical ionization analysis</td>
</tr>
</tbody>
</table>


OPTIX Software - Easy to use software with advanced process applications for plasma gas monitoring of vacuum – can be purchased with spectrometer head alone

1. Connect / Disconnect Remove Optix sensor
2. Optix Device settings
3. Turn the power supply on. Power supply settings.
4. View Spectrum plot.
6. Clear triggers.
7. Trigger settings
8. Species table.
9. Species doughnut.
10. Trigger settings.
11. Species Table.
12. Species doughnut.
13. Species bar chart.
14. The current total pressure reading.
15. Species trend view.
16. Trigger status.
OPTIX Software – Highly refined with the following features included

- Quantitative gas analysis down to 2 ppm (Pressure gauge added for QGA)
- Gas mixture balance – up to 8 gases – can be selected
- Process gas tracking with trigger / alarm outputs
- Full spectrum view 200-850nm, control of integration time for sensitivity adjustments
- Tuneable spectrum view – more focussed range
- Automatic gas peak detection – gas auto indentification database can be adjusted to incorporate additional un-common peaks of specific interest
- Leak detection mode for any gas
- Process water tracker with triggers / alarm outputs
- Chamber pump-down tracker with triggers / alarm outputs
- Vacuum switch to prevent accidental operation at atmosphere
- In-built vacuum pressure reading
- Control of plasma generator to tune power parameters
- Multiple sensor monitoring
- Multiple language display options – English, Japanese, Chinese, French, Spanish
Software Spectrum View - spectrum displays the constituent species of the plasma

The various gas peaks are automatically identified – as only common gases present in a vacuum are identified, this is a much more accurate feature than other spectral identification methods. The auto identification database can be edited.
Software Gas Tracking View
OPTIX Software – an unlimited number of gas species can be monitored via the trend-line feature
OPTIX RPGA Spectrum Interpretation

- A single leak can emit multiple emission lines showing the exact composition
- OPTIX automatically identifies the species which makes interpretation easier
**OPTIX** gas measurement during heating phase of an Arc based hard coating cycle

- Outgassing of species during heating phase prior to coating
- Comparison of outgassing between different tools

![Graphs showing gas measurement over time](image)

- **N2+**: Pressure c. $1 \times 10^{-5}$ mbar
- **CO2**
- **OH**
OPTIX gas measurement during reactive sputtering, comparison with high pressure RGA

Reactive sputtering – process pressure 4E-3 mbar
**TiN deposition cycle – monitoring film growth**

- **Cycle with no TiCl₄**
- **Cycle with TiCl₄**
- Difference is due to N₂ consumption during film growth
- Film growth ends

**Graphs:**
- Top graph: Intensity vs. Time
- Bottom graph: Intensity vs. Time

**Notes:**
- Average of 9 cycles
- TiN deposition cycle
- Monitoring film growth
- TiCl₄ flow
- N₂+H₂ flow through the plasma source
- Ar flow through the foreline
- RF plasma

**References:**
- O. Zabeida, S. Woodward-Gagne, L. Martinu, Polytechnique Montreal
OPTIX for ALD Deposition cycle monitoring

Dr. Richard Potter and Ben Peek

TiO$_2$ deposition cycle monitoring

Precursor injection

ALD reactor

c. 100 mTorr

Heated 90°C

Titanium isopropoxide (TTIP)

Mechanical pump

Water

OH

CH

OPTIX

OPTIX

OPTIX
ALD Deposition cycle monitoring

TiO$_2$ deposition cycle monitoring

- Precursor A – Water vapour
- Precursor B - Titanium isopropoxide (TTIP)
- Water observed via OH (309.6 nm) and TTIP via CH (387 nm)
- 25s cycle time, 2000 cycles, 13 hours for complete process

Titanium isopropoxide (TTIP)

$$\text{TiO}_2$$

Emission intensity (a.u.)

Time (s)

Water

OH

TTIP

CH
OPTIX for ALD Deposition cycle monitoring

- An increase in CH observed as process progresses
- The amount of OH observed after TTIP injection decreases with time
- OH observed after TTIP likely indicator of TTIP/Surface reaction by-products

- Precursor A – Water vapour
- Precursor B - Titanium isopropoxide (TTIP)
- Water observed via OH (309.6 nm) and TTIP via CH (387 nm)
- 25s cycle time, 2000 cycles, 13 hours for complete process

Start (cycles 1-20)
Mid (cycles 100-120)
End (cycles 1900-1920)
OPTIX has two possible power modes DC & Pulsed Mode

Hydrocarbon contamination prevention via patented pulsed power mode

DC mode operation results in eventual contamination of the sensor’s electrodes – resulting in unstable operation

Pulsed mode operation is able to continuously “sputter clean” the electrodes resulting in stable operation over time

PULSED MODE is recommended for all process with high levels of volatile species – ALD, CVD, MOCVD, OLED
OPTIX Pulsed Mode
Organics sensitivity enhancement

OPTIX plasma generation by DC power mode of 200, 500 & 1500 μA compared with pulsed DC at 1500 μA

- DC mode operation can result in weak sensitivity or even suppressed readings for detection of high concentrations of hydrocarbons.
- **OPTIX pulsed mode** significantly increases detection sensitivity of large organic molecules.
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**Signal change after injection of TTIP**

- **OH (309.6 nm)**
- **CH (387 nm)**
- **H (656.5 nm)**

**Sensor electrodes**
- DC mode
- Pulsed mode

**Pulse mode operation**

Very weak CH reading from TMA with DC mode – CH reaction by product not detected

Strong CH reading from TMA detected with pulsed DC mode - CH from methane reaction by product detected
Sensor over the 2+ day deposition cycle displays variations in the process over a longer period which aren’t present for small timeframes.
**OPTIX for outgassing measurement during carbon sputter coating process**

Courtesy of CERN Vacuum Surfaces and Coatings Group

Carbon sputtered coating deposited on particle accelerator inner surface to reduce secondary electron yield
Deposition pressure at **1.1E-1 mbar**
Performance of coating is sensitive to the presence of H outgassing from the magnetron environment
Objective to monitor H outgassing during the deposition

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**Spectrometer saturated**

**Water vapour - 309.6 nm (OH), 18 AMU**

Magnetron turned off and on – OPTIX detects moisture increase as gettering is stopped – RGA not sensitive enough to ‘see’ the event.

Moisture condenses on the surfaces of the RGA differential pumping set and does not reach the detector
OPTIX for Web Coating

Pump down monitoring, Web material plasma pre-treatment characterising, and AlOx magnetron sputter deposition monitoring

- Roll-to-roll deposition of reactively sputtered AlOx onto 125µm PET
- Optix sensor teed with a differentially pumped RGA
**OPTIX** monitoring plasma pre-treatment of plastic web based substrate

- Moisture liberation measured at different treater powers
- OPTIX can also compare moisture liberation from different materials
OPTIX for reactive sputtering of AlOx on roll-to-roll web coater

Reactive sputter characterisation

Ar signal interaction with O2 flow

CO2 observed when the target becomes poisoned – web damage

Target poisoned

Target de-poisoned

Stable feedback control

O2 controller auto-tuning mode

Unstable feedback control

CO2 observed when the target becomes poisoned – web damage
OPTIX CO2 monitoring to determine reasons for CO2 liberation in poisoned mode

- Web speed varied to determine the source of the CO2
  - No influence = cathode
  - Influence = web
- Strong influence of web speed observed
- Inverse effect on O2 observed
OPTIX for a reactive ion etch process

Detection of reactive ion etching effluent in the process backing line

Processing chamber

Etch head

CF4

Ar

Optix

N2 purge

Pressure 4E-2 mbar

Roughing pump
**OPTIX for an etch process end point detection**

End point level

Etching occurring

Etching chamber plasma on

Etching occurring

Graph showing etching process with CO2 and O2 etch conditions.
OPTIX for atmospheric chemical sensing

- Base pressure with valve closed $1 \times 10^{-2}$ mbar
- All tests at 300 µA DC plasma sensor power
- Optix plasma generator and sampling system heated to 50°C
- Solvent container not heated

Overlay comparing the evaporation rates of DEC, IPA and Acetone. Results are consistent with their respective vapour pressures.
Hardware Configurations Options

Standard OPTIX package, plasma generator with power supply (DC as standard, pulsed DC as an option) with Spectrometer head and OPTIX software package / cables

Plasma generator with power supply (DC as standard, pulsed DC as an option) and cables – generates an intense plasma over a wide pressure range – can link to Speedflo or other control platforms

Standard OPTIX package, with optional optical fiber link between sensor and spectrometer – increases flexibility of the package – use items separately

Spectrometer head with OPTIX software package – take advantage of the OPTIX software suite to manage your plasma monitoring and take advantage of the communication and trigger facilities
Size & Communication Options

Optix sensor: 300mm x 170mm x 95mm
PSU: 165 mm x 105mm x 55mm
Sensor weight: 2.2kg
Vacuum connection: KF25 flange
Mounting orientation: Any

Communication interfaces
USB
RS232
Ethernet
Digital relay output x 4
Optional PLC communication interface

Software Windows 7, 8 and 10 compatible
Electrical Input voltage: 15V
Input power: 20W typical
Output voltage: 3kV max
Output current: 1.5 mA max

Operating data

Total pressure operating range: 1x10^{-6} mbar – 0.5 mbar
Sensitivity: 50 ppm air in argon at 1.6x10^{-2} total pressure
Spectral range: 200nm – 900nm
Update speed: 5ms – 5 seconds (depending on sensitivity selected)
Total pressure measurement: Integrated (1x10^{-6} mbar – 1x10^{-2} mbar measurement range)
Electronics maximum operating temperature: 40°C

- Much simpler and smaller hardware than an differentially pumped RGA arrangement
- Comprises the sensor head, small power supply and a remote PC or machine control
- Software for loading onto PC is provided
OPTIX – making Residual Gas Analysis easy for industrial processes

- Highly robust and easy to use – no filaments to replace & easy to use software
- OPTIX can work at all process pressures – no need to differentially pump unless atmospheric sensing
- Highly mobile – can be carried in a small bag for on-site trouble shooting
- OPTIX is less sensitive to contamination than RGA’s, can be used for ’dirty’ hydrocarbon environments as well as etch, CVD and ALD type processes
- This sensing technique offers a lower cost and lower complexity solution than alternative methods
- Can link directly to Speedflo reactive gas controller or PLC for feedback control