

Monitoring of volatile vacuum species using remote optical emission spectroscopy

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Outline of the talk



- Explanation of the Remote Plasma Emission (RPEM) method
- Gas detection and quantification by RPEM
- Examples of data from ALD, Etching and solvent analysis
- Conclusions





RPGA vs RGA



OPTIX – remote plasma gas analysis (RPGA) Optical method

Quadrupole Residual Gas Analyzers (RGAs)





OPTIX Remote Plasma Gas Analysis RPGA

Vacuum process 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





OPTIX operates in the typical plasma processing pressure range

Easy to use and wide operating range





OPTIX vacuum based applications





OPTIX Plasma Generation

Unlike RGA's OPTIX detector is separated from the chemicals by an optical window - more rugged - detector cannot contaminate

- Purpose designed and patented plasma generation source
- Very wide range of operation Plasma present from 0.5 to 10⁻⁶ mbar
- Fast current feedback control
- Constant current = constant excitation source
- DC mode as standard for 95% of ۲ applications, Pulsed DC for highly contaminating atmospheres
- Atmospheric sampling via simple ۰







Software Spectrum View - spectrum displays the constituent species of the plasma







Quantification of gas levels using RPGA

- The sensor results as displayed in the raw spectrum are **qualitative** due to the interaction of different gases within the vacuum
- Even quantities of a gas are equally likely to be collide with free electrons
- Gencoa have developed a mathematical treatment to accurately calculate gas partial pressure





Quantification - Pressure limitations

- Higher currents give a superior signal to noise ratio but at the expense of upper operating pressure limit.
- Maximum linear operating range can be achieved with a lower current setpoint OPTIX can select the current via the user interface.



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Quantification of gas partial pressures – Plasma Power correction

Correction factor per unit pressure —Power ——Correction factor 6 Correction factor, Power (W) 1 0 10 100 Unit pressure 50000 45000 40000 Emission intensity (a.u.) 30000 20000 15000 15000 10000

3

Power (W)

4

5

6

7

- The power delivered to the plasma generator will modify the emission intensities and hence distort the gas partial pressure measurement
- A correction factor based on the measured power can be applied to the emission to remove this effect

5000

0

1

2



Quantification - Power correction

• The effect of the correction can be clearly seen when compared with a differentially pumped RGA





Quantification of gas levels using RPGA

- Introduction of a >> larger quantity of an additional gas will reduce the likelihood of electron impact on species of a << smaller quantity
- This will have an effect of supressing the emission of these species
- The OPTIX has a correction algorithm for the gas interaction effect to allow accurate quantification of the gas partial pressures





Quantification – Gas interaction

Experimental setup

- The most significant challenge for quantification of the sensor readings is the interactivity of gases
- Without correction the readings are **relative** not absolute
- i.e. increasing partial pressure of one gas will lead to a reduction in the readings of other gases.
- An experimental setup was constructed to investigate this effect and to demonstrate the correction method



Diff. pumped side

High pressure side



Quantification – Gas interaction

- Ar, N2, and O2 were mixed in varying quantities
- Total pressure variation was from 1E-5 to 2E-2 mbar on the high pressure side
- Differentially pumped side was kept below 1E-4 mbar





Quantification – Gas interaction

• Gas interaction effects can be clearly seen on the OPTIX readings resulting in different partial pressure measurements compared to the RGA





Quantification – Accurate gas partial pressure measurements after the gas effect correction algorithm is used

- An algorithm can be used to correct for the interaction effects
- Partial pressures can then be derived





Remote Plasma Gas Analysis Highly Sensitive Optical Method

Which species can be observed?

- Atomic emissions and molecular emissions
- Larger molecules are observed as fragments due to disassociation in the sensor's plasma





Caveats and considerations when using Remote Plasma Gas Analysis -Disassociation

• "Fingerprints" of the original molecule





ALD monitoring experimental setup



University of Liverpool





Dr. Richard Potter and Ben Peek



Precursor detection

NH₃





NH (336 nm) overlapping with smaller N2 peak at 337 nm)



0 920 720 770 820 870 970 1020 -1 Time (s) -3 -5 -5 -10 -7 — NH (336 nm) — H (656 nm)



Precursor detection

NH₃





Precursor detection









Atomic layer deposition precursor monitoring

 $H_3C_AC_H_3$

$$_{1}$$
 CH_{3} TMA
 CH_{3}



Deposition cycle monitoring

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Synchronisation of the CCD capture with the ALD pulse





Deposition cycle monitoring

Synchronisation of the CCD capture with the ALD pulse



A series of CCD spectrum captures is synchronised with each precursor injection

Trigger series of integrations



Deposition cycle monitoring



Synchronisation of the CCD capture with the ALD pulse

• The H maxima of each precursor pulse was recorded





Application Example Atomic layer deposition precursor monitoring Deposition of NbN via PEALD



ALD user, Japan



• Detection of TrisNb via CH, N and H



• Detection of NH3 via N and H



OPTIX Atomic layer deposition precursor monitoring

NbN deposition step





ALD Monitoring – Full process for 2.7 hours

Deposition of NbN via PEALD





Atomic layer deposition precursor monitoring

Deposition of NbN via PEALD

• Sensor is robust of the full 2+ day deposition cycle and displays variations in the process over a longer period





Application Example - Characterising a reactive ion etch process

Detection of reactive ion etching effluent in the process backing line

Processing chamber





CF4 detection (no Ar background)





CF4 detection (Ar background)











- Remote PEM combined with spectroscopy can perform "RGA-like" functions
- Can use this method directly at higher process pressures no need to differentially pump unless atmospheric sensing
- The detector is separated from the vacuum environment hence not affected by hostile chemistry present in the vacuum
- OPTIX is hence 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 is highly robust plasma generator will not contaminate or stop functioning