A quantitative approach to Optical Emission Spectroscopy (OES) monitoring of vacuum processing conditions over a wide pressure range and aggressive environments

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Introduction

- Motivation for the remote plasma emission sensing technique
- Sensitivity of the technique
- Field trials of the sensing technique
- Quantification of the readings
- Conclusions
Typical vacuum deposition and surface treatment pressure ranges

- **Evaporation**
- **Sputtering**
- **CVD / PECVD**
- **Plasma surface treatment**

Motivation for the sensing technique
Overview of the sensing technique

Remote Plasma Emission Spectroscopy

- Original concept used by Mann in 1981(!) for leak detection

Spectrum analysis gives species composition

Vacuum Process

Remote plasma generator

Plasma

Miniature spectrometer
• Fast feedback control of the current allows for a stable plasma to be generated from 1E-6 mbar to 1 mbar

• Overall species excitation is determined by the current setpoint.

• The total pressure reading can be inferred from the voltage feedback
• Nitrogen (391.4 nm) was recorded and the change in signal level when the leak was opened and closed was observed.

• PPM levels were progressively reduced in order to find the PPM detection limit by increasing the argon flow.
The change in signal level below 50 PPM was greater than the noise floor average, therefore 50 PPM can be said to be the detection limit.
The PPM limit at lower pressures may actually be significantly lower due to increased sensitivity at lower pressures.
Case study 1 – Outgassing measurement during carbon coating process

- Carbon sputtered coating
- Deposited on particle accelerator inner surface to reduce secondary electron yield
- Deposition pressure of $1.1 \times 10^{-1}$ mbar
- Performance of coating is sensitive to the presence of H outgassing from the magnetron
- Objective to monitor H outgassing during the deposition

Courtesy of CERN Vacuum Surfaces and Coatings Group
Case study 1 – Outgassing measurement during carbon coating process

Hydrogen - 656 nm, 2 AMU

Initial H value higher due to water vapour disassociation

Magnetron turned off
Case study 1 – Outgassing measurement during carbon coating process

Water vapour - 309.6 nm (OH), 18 AMU

Spectrometer saturated

Magnetron turned off
Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

- Roll-to-roll deposition of reactively sputtered AlOx onto 125µm PET
- Optix sensor teed with a differentially pumped RGA

Courtesy of Emerson and Renwick
Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

- Very large H outgassing – taking significant time to reach steady state
- Other species also observed initially outgassing – OH, CO2, O
- Subsequent power increases cause increased H outgassing and additional settling time
- Consumption of N2 also observed – small chamber leak
Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

Reactive sputter characterisation

Ar signal interaction with O2 flow

Target poisoned

Target de-poisoned

O2 controller tuning mode

Unstable feedback control

Stable feedback control
Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

**RGA comparison – CO\textsubscript{2}**

![Graph showing RGA comparison for CO\textsubscript{2}](image)

**RGA comparison – O / O\textsubscript{2}**

![Graph showing RGA comparison for O / O\textsubscript{2}](image)
Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

- There are discrepancies between 675 nm (Ar) and amu 40 (Ar)
- Gradually increasing RGA signal is spurious as Ar flow is constant
- Variations in the Optix Ar signal are due to interaction with the O2 process gas

### RGA comparison – Ar

Influence of changes in reactive gas flow
Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

RGA comparison – OH / H\textsubscript{2}O

Start of target cleaning
Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

- 656 nm (H) and amu 1 (H) are generally a good match.

- The difference at the start of the trace can be attributed to water vapour disassociating inside the Optix sensor into H, increasing the H reading.
Case study 3 – Atomic Layer Deposition

Deposition of NbN via PEALD

High Energy Accelerator Research Organization (KEK), Tsukuba, Japan

Image and data courtesy of S. Kato

- Detection of TrisNb via CH, N and H

- Detection of NH₃ via N and H
Case study 3 – Atomic Layer Deposition

NbN deposition cycle

[Graph showing species concentration over time with labels: NH₃ + Ar, Plasma on, TrisNb, MFC bleed and purge, Cycle repeats. The species concentration is measured as a percentage of sensor full scale.]
Case study 3 – Atomic Layer Deposition

NbN deposition cycle

2.7 hours
Case study 3 – Atomic Layer Deposition

**NbN deposition cycle**

- Sensor is robust of the full 2+ day deposition cycle
Case study 3 – Atomic Layer Deposition

NbN deposition cycle

• Sensor is robust of the full 2+ day deposition cycle
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.
Quantification - Power correction

- A correction factor based on the measured power can be applied to the emission to remove this effect.

- The power delivered to the plasma generator will modify the emission intensities.
• The effect of the correction can be clearly seen when compared with a differentially pumped RGA
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

![Experimental setup image]

Gas input – Ar, N₂, O₂

Diff. pumped side        High pressure side
Diff. pumped RGA

- Ar, N\textsubscript{2}, and O\textsubscript{2} 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.
Quantification – Gas interaction

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

Ar partial pressure readings compared between RGA and OPTIX
Conclusions

• Remote plasma emission monitoring can be used to provide “RGA-like” capability directly at higher process pressures

• Enhanced sensitivity to condensable species over a differentially pumped RGA

• Robustness demonstrated with contaminating processes

• Quantitative data can be obtained over wide pressure ranges and gas mixtures
Thank you for your attention!

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