A NOVEL SENSOR USING REMOTE PLASMA EMISSION SPECTROSCOPY FOR MONITORING AND CONTROL OF VACUUM WEB COATING PROCESSES

F. Papa¹, J. Brindley², T. Williams², B. Daniel², V. Bellido-Gonzalez², Dermot Monaghan²

¹Gencoa USA, Medina, OH, ²Gencoa Ltd., Liverpool, UK

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### Benefits of monitoring the vacuum

- **Scrappage reduction via contamination detection**
  - Leaks, outgassing from chamber/web
  - Control starts in pre-treatment chamber

- **Real time process troubleshooting**

- **End point detection**

- **Process control**
  - outgassing compensation
  - process stabilisation – short and long term

### Process optimization

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**Why monitor?**

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**gencoa: perfect your process**
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<th>gencoa: perfect your process</th>
<th>Current techniques pro’s and con’s</th>
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<tr>
<td>• Pressure gauge</td>
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<td>– Gives quantitative measure of a change in the system, but no information on nature of the change</td>
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<td>• RGA</td>
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<td>– Gives relative quantitative information about gas composition and partial pressure</td>
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<tr>
<td>– Limited operating pressure $&lt; 5 \times 10^{-3}$ mbar</td>
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<td>– Differential pumping needed for higher pressures</td>
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<tr>
<td>• Expensive</td>
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<tr>
<td>• Limited sensitivity</td>
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<tr>
<td>– Maintenance / calibration</td>
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<td>• Optical Spectroscopy of remote plasma</td>
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<tr>
<td>– Low maintenance and simple setup</td>
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<tr>
<td>– Gives compositional data about gases (semi-quantitative)</td>
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<td>– Need to analyze spectra</td>
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Combining remote PEM with a spectrometer gives “RGA-like” capability.
• Fast feedback control of the current allows for a stable plasma to be generated from 1E-6 mBar to 0.5mBar

• Overall plasma intensity is determined by the current setpoint

• The total pressure reading is inferred from the voltage feedback
• Operates directly at the most common processes pressures

• No spurious readings from differential pump system

• Direct monitoring of the vacuum means that changes are instantly registered

• Significantly cheaper than RGA + differential pump!
• Atomic and molecular emission lines
• Larger molecules are observed as fragments

For example: \((\text{CH}_3)_2\text{CHOH}\) observed as CH, H, O, OH and CO2
Unique lines corresponding to N2

Automated identification
Databases can be used to automatically interpret the spectrum

Example of an air leak:
Automated identification is integrated into the sensor’s software.
Vacuum quality monitoring

- Air pumping
- Water vapour removal

1 mBar

< 1E-5 mBar

Pumpdown Vacuum Quality Analysis

- N2 337nm
- OH 309 nm
- H 656nm
Process pressure = 1x10^{-1} mBar
Ratio of H to Ar was varied during experiment

The hydrogen emission intensity is proportional to the contamination ratio
The Optix sensor is mounted directly to the chamber, close to the magnetron. Water vapour consumption by the magnetron is clearly observed.

The process is also being sampled through a differentially pumped RGA. The consumption of water vapour is not observed by the RGA due to condensation in the pumping system and bellows.
Monitoring of SF6

SF6 is observed by disassociation in the plasma into fluorine.
Water vapor outgassing from the web can be monitored using the sensor. 

Sensor response when adding water vapor to the vacuum

![Graph showing emission intensity vs. wavelength](chart.png)

- Increasing water vapor partial pressure
- OH peak at 300 nm
Sensor response when adding PET to the vacuum

Measurement of water vapor can be used either open-loop or closed loop: i.e. fed back into a controller for regulating treater parameters – useful for magnetron pre-treaters for high speed web processing.
Ion etching end point detection, process pressure 1E-4 mBar

- Increases in O2 pressure indicates organic removal from sample
- Final run displays no variation in O2 – no organic removal so process ends
- OPS sensor 1E-6 mBar l/s – will already be in-situ on chamber
- RGA mass spectrometer 1E-7 mBar l/s
- Dedicated leak checker 1E-9 mBar l/s
Comparison of a differentially pumped RGA and Optix sensor

- **gencoa: perfect your process**
- AC, dual rotatable AlOx deposition

**Graph 1:**
- **Target clean (4 kW)**
- **Poisoned (1 kW)**
- **Clean (4 kW)**
- **Reactive mode (4 kW)**

**Graph 2:**
- **H outgassing from magnetron**
- **Shutter opened**

**Graph 3:**
- **Air leak on oxygen line, bleeding out when MFC is opened**
Emerson and Renwick
GENESIS roll-to-roll coater

- Pre-treatment of a material prior to coating is essential for good adhesion and film properties
- Different materials (and even different batches) can have different requirements for optimum pre-treatment
- On-line monitoring of the species liberated during pre-treatment enables optimisation of the treater parameters
- Scrappage can be reduced via detection of any species that indicate damage is occurring on the material
Plasma pre-treatment of 125 µm Melinex® polyester film

Water vapor removal during plasma pre-treatment

- Intensity (counts)
- Time (s)
- Treater power

OH

0.5 kW 1 kW 2 kW 3 kW 4 kW 5 kW

Time (s)
The effect of web speed on water vapor removal

Intensity (counts)

Time

Web speed

0.5 m/min

0.4 m/min

0.3 m/min

0.2 m/min

0.1 m/min

OH

Plasma pre-treatment of 125 µm Melinex® polyester film
A4 fabric samples were taped to the drum with kapton tape and then fed into the treater zone.
Sample was moved into treater zone and held in a static position during the plasma treatment. The objective was to determine if a reduction in OH could be observed after a period of time – meaning that the water content of the fabric was reducing.
Circled in red are outgassing species that could indicate damage to the fabric during plasma treatment.
Increase in CH level could indicate that the fabric is being damaged by the plasma treatment.
Increases in water vapor (indicated by OH) are seen when the plasma treater is turned on and when the sample fabric passes through the treatment zone.
Data was normalised prior to comparison to account for higher level of water vapour present for the Gabardine sample at the end of pumping.

Results indicate that the Cashmere sample has a higher water vapour content or that the plasma treater is more effective at removing water vapour from the Cashmere sample.
Remote PEM combined with spectroscopy can perform “RGA-like” functions.

Can use this method directly at higher process pressures.

This sensing technique offers a low cost / low complexity solution.

Useful tool for improving process control and for gaining insight into what is happening in the web coater.

Can output “emission” signal to closed loop reactive gas controller.
Thanks to Nick Butcher at Emerson and Renwick for their cooperation.

Thanks for your attention