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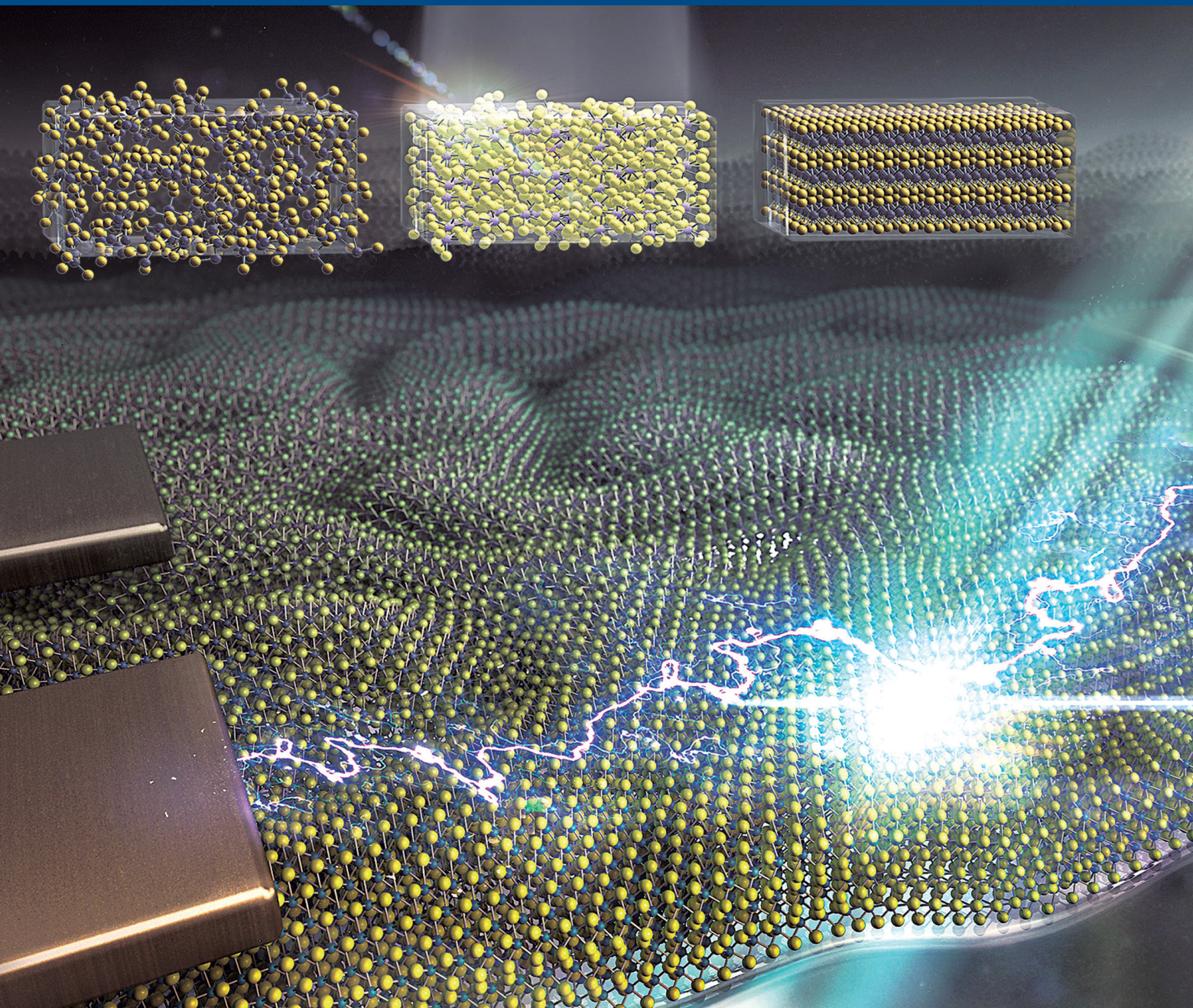

CONTRIBUTED ORIGINAL ARTICLE

Adapted from a manuscript that was presented at the 2025 68th SVC TechCon

Transforming Leak Detection in Vacuum Coating Systems with Remote Plasma Optical Emission Spectroscopy (RPOES)

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In this study, we introduce an alternative leak detection method based on **remote plasma optical emission spectroscopy (RPOES)**. The technique exploits plasma-induced light emission to detect leaks in °real time, enabling both detection and localisation using trace gases such as argon that are relatively inexpensive and readily available. Compared with mass-filter helium detectors, RPOES provides greater robustness, the ability to operate at higher pressures, and reduced maintenance demands. We outline the principles of RPOES, emphasise its advantages in detecting gases commonly linked to vacuum leaks (including water vapor and air), and assess its viability as a practical alternative to helium-based methods. Case studies are presented to demonstrate its effectiveness in identifying leaks across a range of coating applications, with ultimate aim to improved process stability, enhanced coating quality, and lower operational costs.

INTRODUCTION

Ensuring the integrity of vacuum systems prior to deposition and surface treatment is critical, as even small leaks can compromise coating quality, reduce process stability, and result in costly downtime. At present, helium leak detectors based on mass spectrometry and are regarded as the industry standard due to their exceptional sensitivity. However, reliance on helium as a tracer gas presents growing challenges: its cost continues to rise (price doubled over the past decade [1]), long-term availability is uncertain, and the instruments themselves are expensive to manufacture and maintain. Moreover, in many industrial contexts, the level of sensitivity provided is orders of magnitude higher than practically required.

Vacuum system integrity can also be compromised by a range of additional bottlenecks beyond simple physical leaks. These include virtual leaks, where trapped volumes, for example in screw threads or O-ring grooves release gas slowly into the chamber; permeation of gases through materials such as gas lines and seals; and the release of water vapor absorbed on surfaces. Other contributors include organic outgassing from contaminated parts and pumping issues such as oil backstreaming. Each of these factors can degrade vacuum quality, reduce coating performance, and increase the demand for effective leak detection methods to quantify leaks in vacuums.

Leak flow (Q) is defined as

$$Q = \frac{\Delta P \cdot V}{\Delta t} \quad \textbf{(1)}$$

Where ΔP is the change in pressure, V is the volume and Δt is the time taken for the pressure change. Q is typically expressed in mbar·L/s and in SI units Pa·m^3/s.

A number of conventional leak detection methods are employed in industry, but each presents limitations. Rate-of-rise testing (using equation (1)) cannot distinguish the cause of a leak, with leak rates strongly dependent on the initial pump-down conditions. Base pressure monitoring likewise fails to reveal the underlying cause and can take days to diagnose. Dedicated helium leak checkers, while highly sensitive, face rising helium costs and supply chain concerns, with filaments that degrade over time, are prone to contamination, and require regular maintenance and calibration. Residual gas analysers (RGA) based on mass spectrometry allow detailed analysis but require sampling of the vacuum system, involve complex and sensitive components, and are often overkill for the practical requirement. Furthermore, data interpretation can, in some circumstances be non-trivial, as contaminants can share the same mass-to-charge ratios.

To address these limitations, we introduce a leak detection approach based on **remote plasma optical emission spectroscopy (RPOES)**. An early demonstration of plasma-based optical emission for leak detection was reported by Mann [2], who developed a simple detector based on monitoring the light emitted from a Penning gauge plasma. In this configuration, the emission line at 391.4 nm, corresponding to the N_2^+ ion, was selectively filtered and detected, allowing air leaks within a vacuum chamber to be identified. This early work established the principle that plasma-induced optical emission could serve as a sensitive indicator of gas ingress, laying the foundation for more advanced remote plasma optical emission spectroscopy (RPOES) techniques.

Such a system is described in this publication to detect gases in real time, providing partial pressure information. It is well suited to identifying gases commonly linked to vacuum leaks, such as water vapor and air, and can be used in conjunction with a trace gas, making it a practical alternative to established methods.

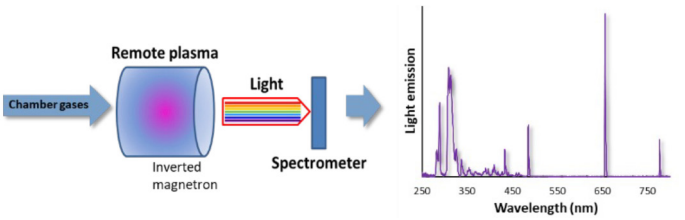

Fig. 1 – RPOES technique.

EXPERIMENTAL AND RESULTS

RPOES technique

The RPOES technique operates by generating a small remote plasma within a dedicated sensor mounted to the process vacuum chamber. In this configuration, species present in the chamber are ionised and excited within the sensor plasma, producing characteristic optical emission lines. The resulting light is transmitted to a spectrometer, where both the wavelength and intensity of the emission features are analysed. Identification of the emitting species is achieved by cross-referencing the observed lines with established reference data, including the NIST Atomic Spectra Database [3] and Pearse and Gaydon's *Identification of Molecular Spectra* [4]. By quantifying the intensity of the identified peaks, the partial pressures of the corresponding gases can be determined, enabling both qualitative and quantitative assessment of the vacuum composition.

The sensor used employed an inverted magnetron configuration to generate the plasma. A cylindrical cathode surrounded a rod anode biased up to 3 kV, while a surrounding ring magnet provided confinement of electrons close to the anode, sustaining a stable Penning discharge. The light emitted from the plasma was collected through a sapphire viewport and collimating optics, then directed into a compact broad band spectrometer. This configuration enabled analysis across a broad spectral range, from the ultraviolet to the near infrared, thereby capturing multiple emission peaks simultaneously rather than being limited to a single filtered wavelength. Its basic principle is shown in Fig. 1.

Current regulation of the discharge was used to maintain monotonic behaviour of plasma current (emission intensity) with respect to increasing partial pressure, providing a stable calibration basis. The operating range of the sensor was established from 10^{-6}mbar to 0.5 mbar, covering the regime relevant for industrial vacuum processes and leak detection. This is illustrated in Fig. 2.

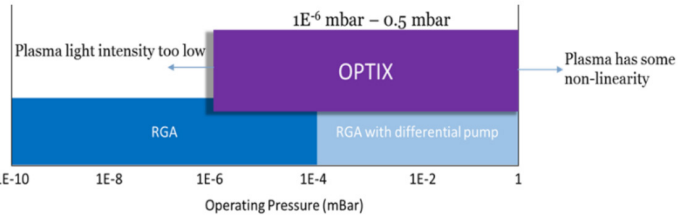

Fig. 2 – RPOES device operating pressure range.

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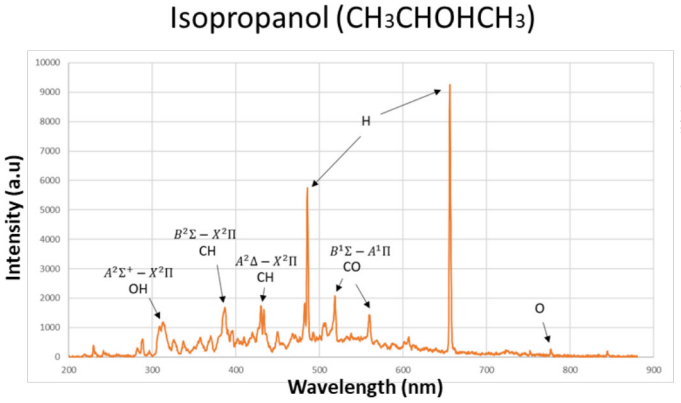

Fig. 3 – OES spectrum of Isopropanol.

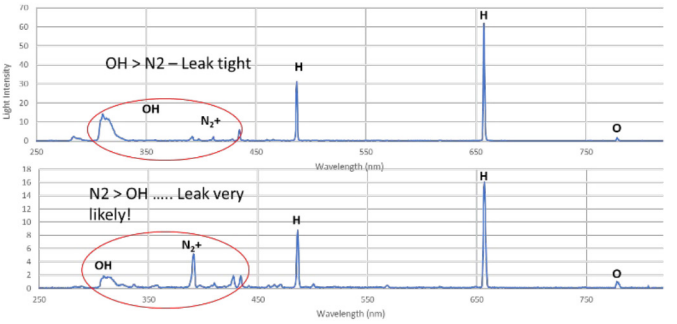

Fig. 4 – Water vapour and air leaks.

An important feature of the RPOES technique is that the plasma dissociates larger molecules into their smaller constituent fragments. These fragments emit light at well-defined wavelengths characteristic of their elemental or molecular identities. Consequently, the recorded spectrum contains a series of prominent and easily identifiable peaks, which can be used to infer the presence of the original parent molecule with high confidence. Shown in Fig. 3 is a spectrum of isopropanol.

Application Examples

The case studies reported in this work, had a measurement time (light acquisition) of 1 s unless stated otherwise. The device was operated in HDR mode, where the acquisition time was split into segments of differing lengths and reconstructed into one spectrum. This algorithm enhanced measurement quality, allowing small signals to be measured in detail. Since the limiting factor for measurements is the amount light accumulation, for some applications, an acquisition time of < 100 ms is sufficient.

Application examples are presented to demonstrate the capability of RPOES for real-time gas identification and quantification in vacuum systems. These results illustrate its effectiveness for leak detection, leak characterisation, and process monitoring.

Air leaks and water content

Fig. 4 shows representative RPOES spectra demonstrating the ability to distinguish between water vapour outgassing and atmospheric leakage. In a clean, leak-tight system, the spectrum is dominated by OH and H emission lines (water), with only a minor contribution from N_2^+. Under these conditions, the intensity ratio N_2^+/OH remains below one (N_2^+/OH < 1). A general rule of thumb is that below 10^{-2} mbar total pressure, nitrogen should not be present in significant quantities.

In contrast, spectra obtained during a leakage event exhibit a large N_2^+ proportion relative to OH, such that the N_2^+/OH ratio exceeds N_2^+/OH > 1. This shift provides a simple and robust diagnostic: systems where nitrogen emission exceeds that of OH can be classified as exhibiting an air leak, while those with OH-dominated spectra are indicative of water vapour outgassing and/or water leaks.

The utility of this ratio can be extended to quality control (QC). In this specific example, assessment of vacuum chambers leak tightness was quantified to a QC specification. The principle is show in Fig. 5 using partial pressure traces of OH and N_2, measured by RPOES in RGA mode. N2 sum in Fig. 5 is the combination of both N_2 and N_2^+ partial pressures, to give a full representation of the nitrogen concentration throughout the pumpdown as the ionisation levels of nitrogen change. The RPOES device was attached directly to the chambers.

An acceptable partial pressure of N2 sum (air) was established for QC purposes in manufacturing. Chambers passing QC, OH emission remains greater than N_2, with nitrogen sufficiently low (Fig. 5(a,b)), corresponding to acceptably leak-tight operation. In contrast, chambers falling outside the QC specification show spectra where N_2 exceeds OH. A small leak pro-

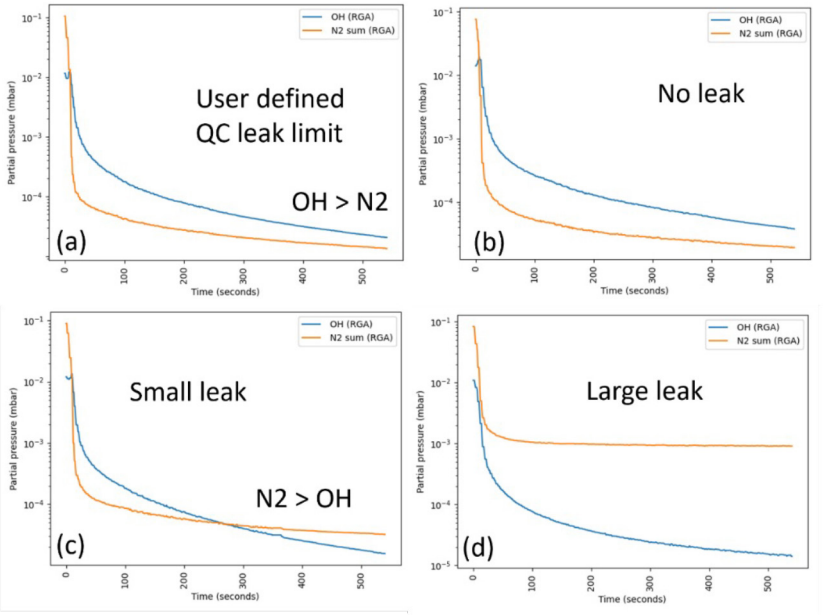

Fig. 5 – Vacuum chamber quality control partial pressures of Nitrogen and OH (water vapour)

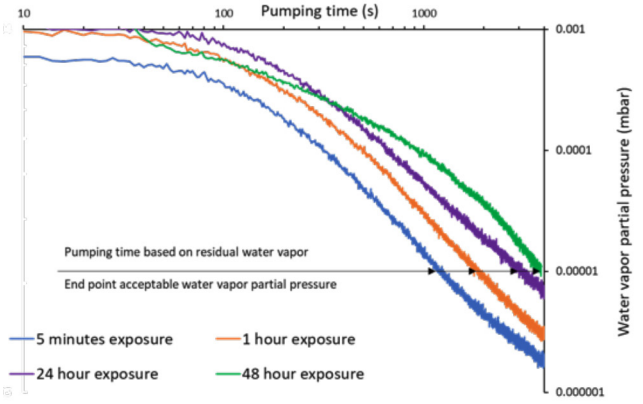

Fig. 6 – Water vapour partial pressures for differing levels of atmospheric exposure.

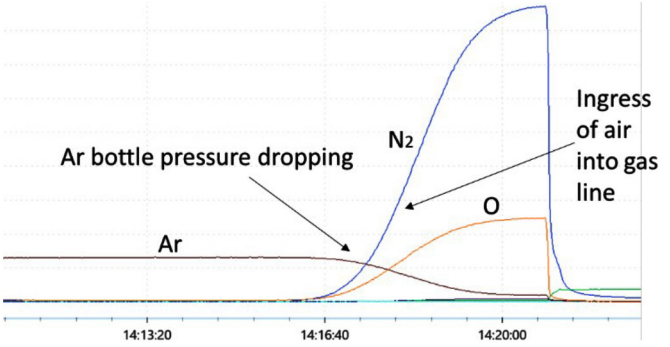

Fig. 7 – Argon, Nitrogen and Oxygen traces for an emptying argon gas line.

duces a gradual crossover between the traces (Fig. 5(c)), while a large leak results in persistently high nitrogen concentration dominating the signal (Fig. 5(d)).

Together, these results establish the N_2/OH signal balance as a rapid and quantitative diagnostic for vacuum integrity, providing a straightforward pass/fail criterion for QC inspection of vacuum chambers, without a tracer gas or mass spectroscopy.

RPOES can also be applied to monitor water vapour desorption following atmospheric exposure, providing a means to optimise pump-down and degassing cycles. Fig. 6 shows the evolution of water vapour partial pressure during pump-down after a vacuum chamber was exposed to atmosphere for different durations ranging from 5 minutes to 48 hours. As expected, longer exposure times result in higher initial water vapour concentrations and extended degassing times before reaching an acceptable base level.

A key advantage of this approach is the ability to define a quantitative end-point criterion for residual water vapour. By monitoring the partial pressure directly, the pump-down stage can be terminated, and the process can be started, once the water concentration has reached the required threshold. This ensures that contaminant limits are met while avoiding unnecessarily long pumping cycles.

Such real-time end-point detection provides a practical route to reducing cycle times and improving process efficiency, while maintaining strict control over vacuum cleanliness

Contamination detection

In addition to system leak detection, RPOES enables the identification of contamination within a system, which cannot be diagnosed by conventional leak testing. Fig. 7 shows a case where an argon process gas line became contaminated with air as the supply bottle pressure dropped. Under these conditions, mass flow controller (MFC) feedback reported no anomalies, and pressure readings remained consistent.

The in situ RPOES spectra, however, revealed a clear ingress of nitrogen and oxygen into the gas line. As the argon supply declined, the N_2 and O emission signals increased in the same period as the argon signal decreased. This directly indicated the replacement of process gas with atmospheric air. Importantly, this situation does not correspond to a system leak, but rather a leak within the gas feed, meaning that in situ gas monitoring is the only way to observe the problem in real time.

RPOES can also be applied to monitor the outgassing behaviour of materials in vacuum environments. In this example the effectiveness of barrier coatings on polymers was quantified using this technique.

Fig. 8, compares the evolution of gas species released from A polymer with and without a silicon nitride (SiN) barrier layer during thermal exposure. In the uncoated polymer (Fig. 8(a)), significant outgassing of CO_2, OH, and N_2 is observed as the sample is heated, with peak concentrations occurring around

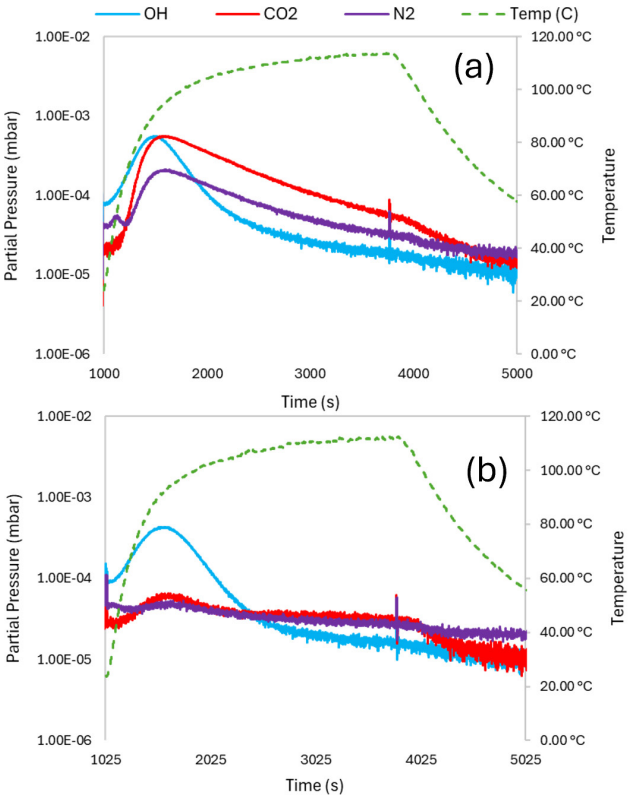

Fig. 8 – Outgassing of polymers with (b) and without a barrier coating (a).

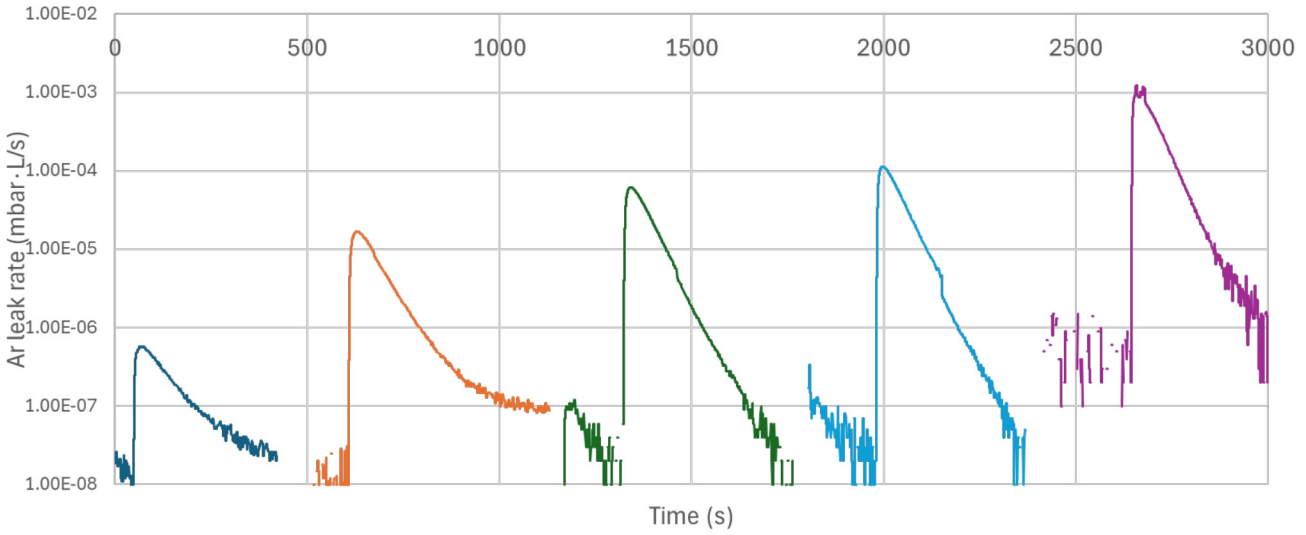

Fig. 9 – Example leak detection experiments using RPOES. All values are stated as the He equivalent.

1500 s. By contrast, the SiN-coated polymer (Fig. 8(b)) shows a marked reduction in CO_2 and N_2 partial pressures, with overall gas release suppressed by over an order of magnitude compared to the uncoated sample. The OH signal remains the dominant feature but is also reduced in magnitude.

These example highlights a fundamental feature of RPOES: the ability to continuously monitor process gases and identify contamination events that would remain invisible to conventional diagnostic tools. This case study demonstrates how a quantitative, real-time method to assess barrier coating performance was performed using the technique, which allowed differences in gas evolution between coated and uncoated polymers. The technique therefore offers a valuable diagnostic for evaluating and optimising materials in coating and packaging applications.

RPOES as an alternative to conventional mass filter-based helium leak checkers

Leak detection experiments were carried out using the RPOES system connected to a vacuum chamber. A dosing valve was fitted to the chamber to introduce controlled leaks under reproducible conditions. In addition, a calibrated reservoir leak (10^{-6} mbar·L/s) was used to provide a reference, enabling conversion of the measured argon signal into quantitative leak rates.

For test measurements, argon was introduced at the chamber opening to simulate leak events. The resulting optical emission signals were recorded by the RPOES device, and data were collected under varying leak conditions to evaluate sensitivity, response time, and repeatability. Fig. 9 shows these results.

Comparisons were made between different leak magnitudes, with emphasis on the system's ability to resolve small leaks and distinguish between signal levels. This approach allowed direct correlation between leak rate and the optical emission response, thereby establishing the calibration basis for quantitative leak detection using RPOES.

Leaks in the range of 10^{-3} – 10^{-7} mbar·L/s were recorded in this example. Further work is needed for full calibrated measurements.

DISCUSSION

The results and case studies demonstrate that remote plasma optical emission spectroscopy (RPOES) offers several clear advantages compared with established helium-based and mass-spectrometric leak detection methods. A primary benefit is lower operational cost. Unlike helium leak detectors, RPOES requires no dedicated pumping or sampling system and avoids reliance on increasingly expensive and supply-constrained helium. The use of inexpensive and widely available gases, such as argon makes the technique particularly attractive for industrial settings where routine leak checking is required.

Another key advantage is measurement speed. Because the RPOES device is in contact with the vacuum environment, there is no lag associated with sample extraction or transfer. Spectral information is delivered to the detector at the "speed of light," enabling rapid identification of leaks and near real-time response during process monitoring.

The technique also provides reduced maintenance demands compared with conventional systems. Helium leak checkers and residual gas analysers (RGA) often rely on fragile filaments or complex ion optics that degrade over time, requiring frequent calibration and servicing. In contrast, the RPOES technique described in this work employs a robust plasma generation with minimal consumable components, ensuring stable long-term operation.

Portability is another strength of the method. The compact design of the RPOES device implementation allows the device to be easily hand-carried and positioned at different points on a vacuum system, in contrast to bulky helium leak detectors that require pumps, carts and storage.

Importantly, RPOES provides a reliable measurement of true gas concentrations within the chamber. Because gases are excited and detected directly in situ, the measurement is not influenced by sampling artefacts. This results in faster signals, more accurate identification of the leaking gas, and the ability to capture subtle features such as virtual leaks or slow outgassing phenomena that might otherwise be masked.

Taken together, these advantages position RPOES as a practical and scalable alternative to helium-based leak detection, particularly for industrial coating and surface treatment processes where ultimate sensitivity is less critical than cost, speed, and robustness.

CONCLUSIONS

This work highlights the potential of remote plasma optical emission spectroscopy (RPOES) as a powerful tool for quantifying vacuum integrity in real time. The technique is inherently non-invasive, relying on the detection of characteristic optical emissions from gases directly within the vacuum environment, and is capable of operating across a wide pressure range from 0.5 mbar down to 10^{-6} mbar. Data collection speeds of under 100 milliseconds enable rapid results and near-instantaneous feedback during leak testing and process monitoring.

Importantly, RPOES can be employed as a standalone leak detection solution, removing the dependence on helium tracer gas and its associated costs and supply chain challenges. Preliminary studies indicate that leak rates as low as 10^{-8} mbar·L/s are within reach using argon, with further optimisation ongoing. Combined with its compact, robust design and reduced maintenance demands, RPOES offers a scalable alternative to conventional methods for industrial applications.

Overall, RPOES provides a versatile, cost-effective, and rapid approach for maintaining vacuum integrity, with clear advantages for coating, deposition, and other vacuum-based processes where both reliability and operational efficiency are critical.

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Marcus Law MPhys, PhD is an Applications Development Engineer at Gencoa, working within the R&D department. He holds a PhD from the Fusion Centre for Doctoral Training at the University of Liverpool, awarded in 2024. In his role, he is responsible for the experimental performance characterisation and data collection for sensing products, leading industry trials, and product demonstrations, and providing customer support, including training and troubleshooting.

Joe Brindley completed his PhD in feedback control algorithm design at the University of Strathclyde in 2011. Joe joined Gencoa Limited in 2012 and has had responsibility for development of their vacuum sensing and control products. Since 2025 he has held the role of Technical Director with overall responsibility for the company’s R&D and product design activities.

Benoit Daniel MSc and BSc(Hons) from St Joseph College, Dijon (France) and Hogeschool Brabant, Breda (Holland) in 2000-2003 in International Project Management in Computer Science and Networking and Software Engineering. He joined Gencoa in 2003 and is currently responsible for the development and production of control & sensing solutions.

Dr Oisín Boyle is a data scientist working at Gencoa to produce AI tools for propelling the vacuum coating sector towards industry 4.0. Having completed his BSc Physics and Music degree with First Class Honours in 2019 at Cardiff University and MSc in Applied Mathematics at Imperial College London in 2020, Dr Boyle completed his PhD in 2025 at the University of Liverpool with a thesis entitled “Block Sparse Bayesian Learning with Applications to Spectral Unmixing for Plasma Optical Emission Spectra”. The research of this PhD comprised analysis of the spectra produced by the Gencoa Optix. Dr Boyle also has experience in high-performance computing, having undertaken training at Hartree STFC and used the University of Liverpool’s supercomputer. Dr Boyle was also a finalist for the Siemens’ AI Challenge in 2021, and a recipient of the Society of Vacuum Coaters Foundation Jim Colbridge Memorial Scholarship.