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Microplasma Spectroscopy for ppm-Level O_2 in Advanced-Reactor Cover Gas

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1. Introduction

Maintaining a tightly controlled oxygen level in the cover-gas of advanced reactors is essential: in HTGRs, oxygen oxidizes graphite and degrades thermo-mechanical integrity [1]; in MSR systems, drift toward oxidizing conditions accelerates corrosion and materials degradation [2]. For SMRs, in-situ monitors must be compact and low-burden to install, making vacuum-based or alignment-sensitive instruments not ideal for deployment on a cover-gas system. Prior work on microplasma spectroscopy for noble-gas monitoring demonstrated a low-cost, real-time alternative suitable for SMR cover-gas systems and overcame the practical limitations of conventional approaches. Our microplasma spectroscopy sensor, previously validated for Xe/Kr quantification, has been extended to measure ppm levels of oxygen (O_2) in Ar and He matrices [3]. This extension utilizes the same robust protocols for discharge stabilization, normalization, and calibration that previously enabled continuous noble-gas tracking.

2. Methods

A microplasma spectroscopy system was developed to monitor oxygen in a helium (He) and argon (Ar) matrix. As illustrated in Fig. 1, flow rate of high-purity (99.999%) He (or Ar) and a mix gas (He (or Ar) and [O_2] 10 ppm) were controlled via two mass flow controllers (MFCs). The O_2 concentration delivered to the X-Sentry cover-gas sensor is verified in real time using a reference oxygen analyzer (Systech Illinois, EC900) installed on a downstream bypass.

The cover-gas sensor consists of a microplasma discharge chamber, a DC power supply, and a UV-Vis spectrometer. The DC source, operated in constant-current mode (up to 6 mA with a compliance of ≤ 6 kV), sustains a stable glow discharge between two tungsten electrodes. The resulting microplasma generates characteristic emission from the gas mixture; light collected through an optical window is coupled directly to the UV–Vis spectrometer (Avantes ULS2048CL-EVO) for analysis.

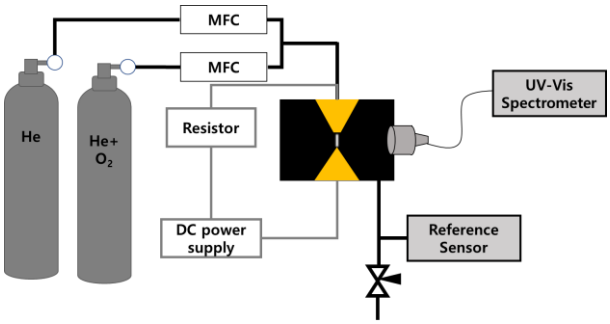

Fig. 1. Experimental setup for sensor calibration for oxygen quantification.

For calibration, spectra were recorded while varying the flow rates of 99.999% He (or Ar) and a 10 ppm O_2 in He (or Ar) mixture to realize a series of O_2 setpoints (typically 0–10 ppm) at a fixed total flow. After each adjustment, the downstream reference oxygen analyzer was monitored, and data acquisition began only after the oxygen analyzer reading stabilized. Emission spectra were acquired with an integration time of 1 s. Oxygen was quantified using the O I 777.2–777.5 nm triplet, and ratiometric normalization to a strong matrix line was applied to suppress drift: He matrix: $R=I_O/I_{He}$ (587.6 or 706.5 nm); Ar matrix: $R=I_O/I_{Ar}$ (852.1nm). Calibration curves were obtained from linear regression of O concentration versus R.

For in-situ monitoring, the sensor system connected to an Ar glovebox (KOREAKIYON) runs continuously under constant-current discharge (6 mA). O 777 nm signal and its conversion to O_2 concentration via the established calibration. Measured O_2 concentrations were compared with the electrochemical oxygen sensor, which is connected to the glovebox.

3. Results and Discussion

The O (777 nm) to Ar (852 nm) normalized intensity showed a clear linear response across 0–5 ppm. The fit quality was high ($R^2 \approx 0.972$) with point-wise relative errors of roughly 4–14% (Fig. 2). Early trials at low total flow (<500sccm) exhibited an apparent oxygen baseline caused by oxygen release from metal surfaces (oxide/adsorbate desorption and sputter-induced release) when the plasma was ignited. Raising the total flow to 1000 sccm minimized residence time and surface re-entrainment, stabilized the baseline, and yielded reproducible calibrations.

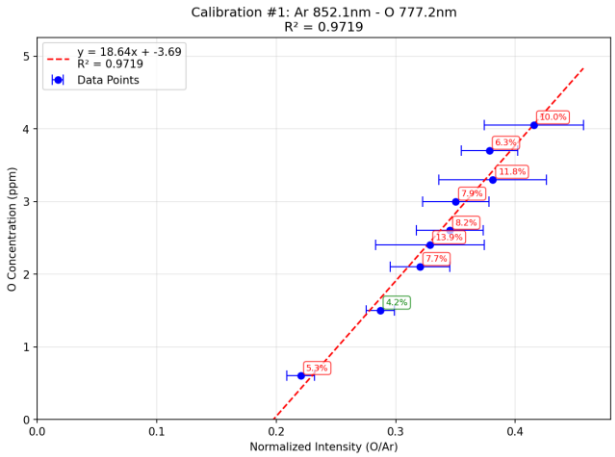

Fig. 2. Calibration of the microplasma spectroscopy oxygen sensor in Ar using O I 777.2 nm normalized to Ar I 852.1 nm.

Connected to the Ar glovebox and operated in constant-current mode (6 mA), the sensor tracked oxygen in real time while the plasma chamber experienced noticeable pressure/flow fluctuation. The ratiometric processing suppressed most intensity swings, and the inferred oxygen level stayed near ~2 ppm, in agreement with the oxygen sensor connected to the Glovebox. Despite the non-ideal pressure stability, these results confirm the feasibility of continuous O_2 monitoring on a real cover-gas loop.

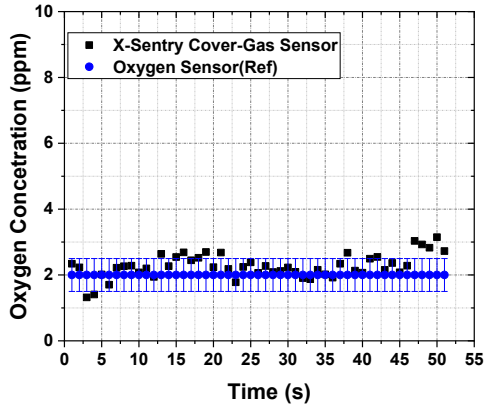

Fig. 3. Real-time O_2 monitoring in an Ar glovebox: X-Sentry cover-gas sensor (black) vs. electrochemical reference (blue).

4. Conclusion

We demonstrated a compact, vacuum-free microplasma spectroscopy sensor that quantifies ppm-level oxygen in helium and argon cover gas. Using the O I 777.2–777.5 nm triplet with matrix-line normalization (Ar 852.1 nm; He 587.6/706.5 nm), the sensor produced a stable, linear response in Ar over 0–5 ppm ($R^2 \approx 0.972$). In the Ar glovebox, agreement with the reference near ~2 ppm was maintained despite pressure/flow fluctuations, indicating feasibility for continuous cover-gas monitoring of 4th generation SMRs. Future work will (i) complete He-matrix calibration and cross-matrix transfer, (ii) manage potential self-absorption at higher oxygen concentration, and (iii) evaluate long-term stability and radiation/thermal endurance with packaged hardware for reactor-relevant environments.

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