



NCCA VS

22nd February 2018

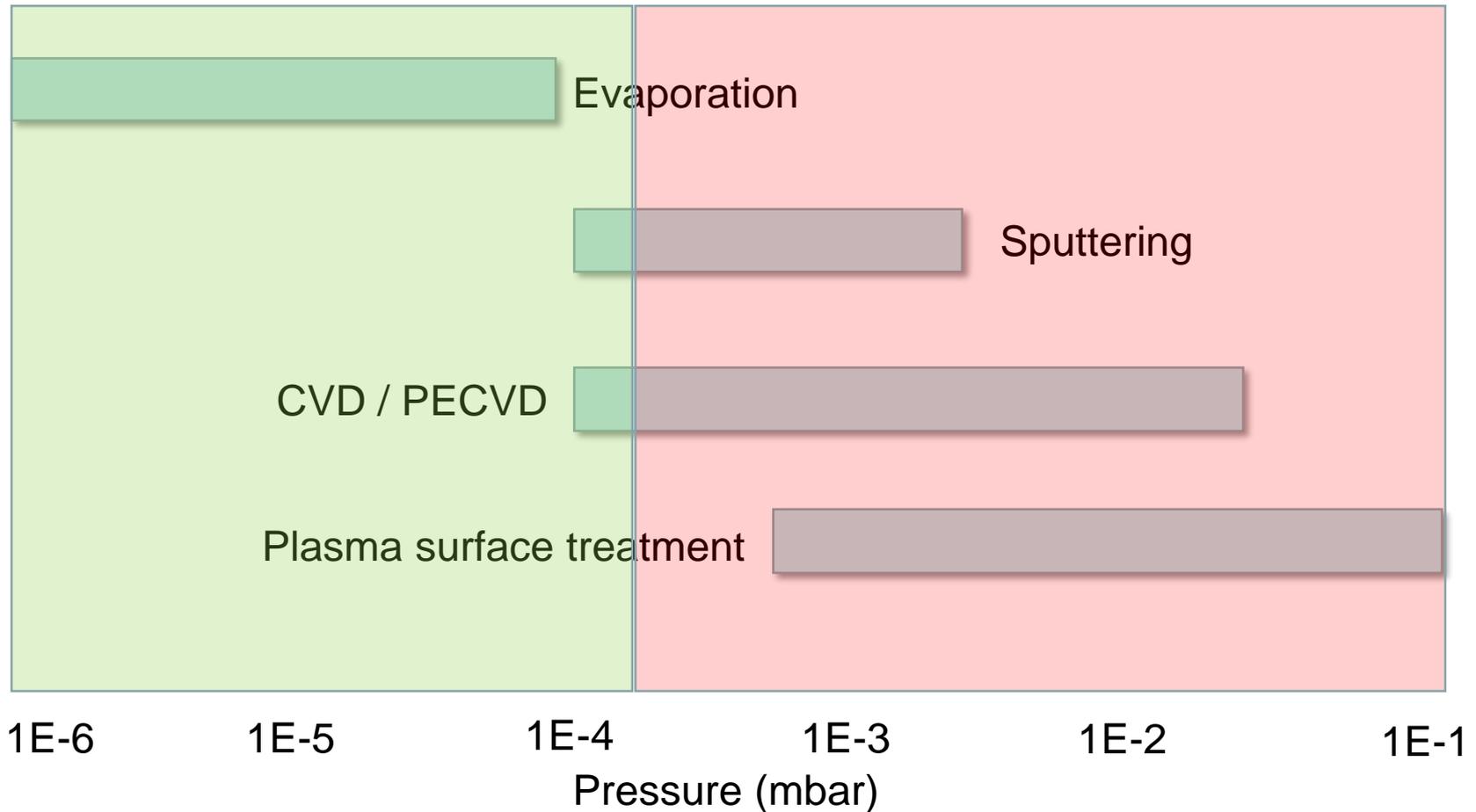
A quantitative approach to Optical Emission Spectroscopy (OES) monitoring of vacuum processing conditions over a wide pressure range and aggressive environments

Frank Papa, Joe Brindley, Benoit Daniel, Victor Bellido-Gonzalez

Genco USA, Medina, OH
Genco Limited, Liverpool, UK

- Motivation for the remote plasma emission sensing technique
- Sensitivity of the technique
- Field trials of the sensing technique
- Quantification of the readings
- Conclusions

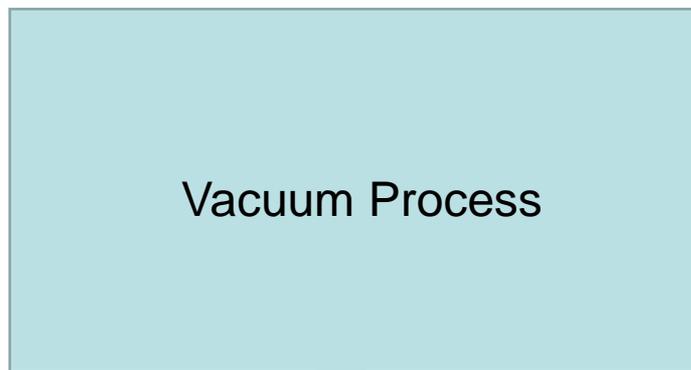
Typical vacuum deposition and surface treatment pressure ranges



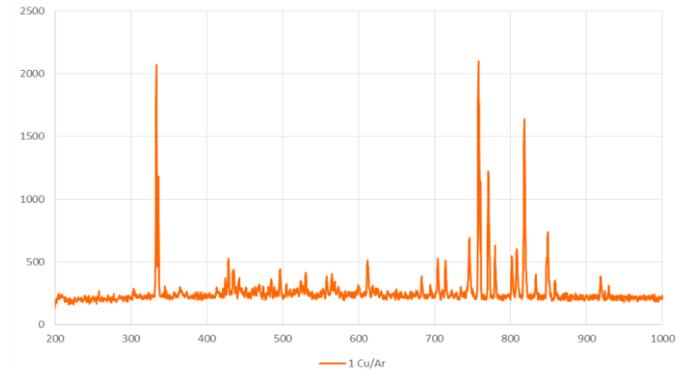
Remote Plasma Emission Spectroscopy

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

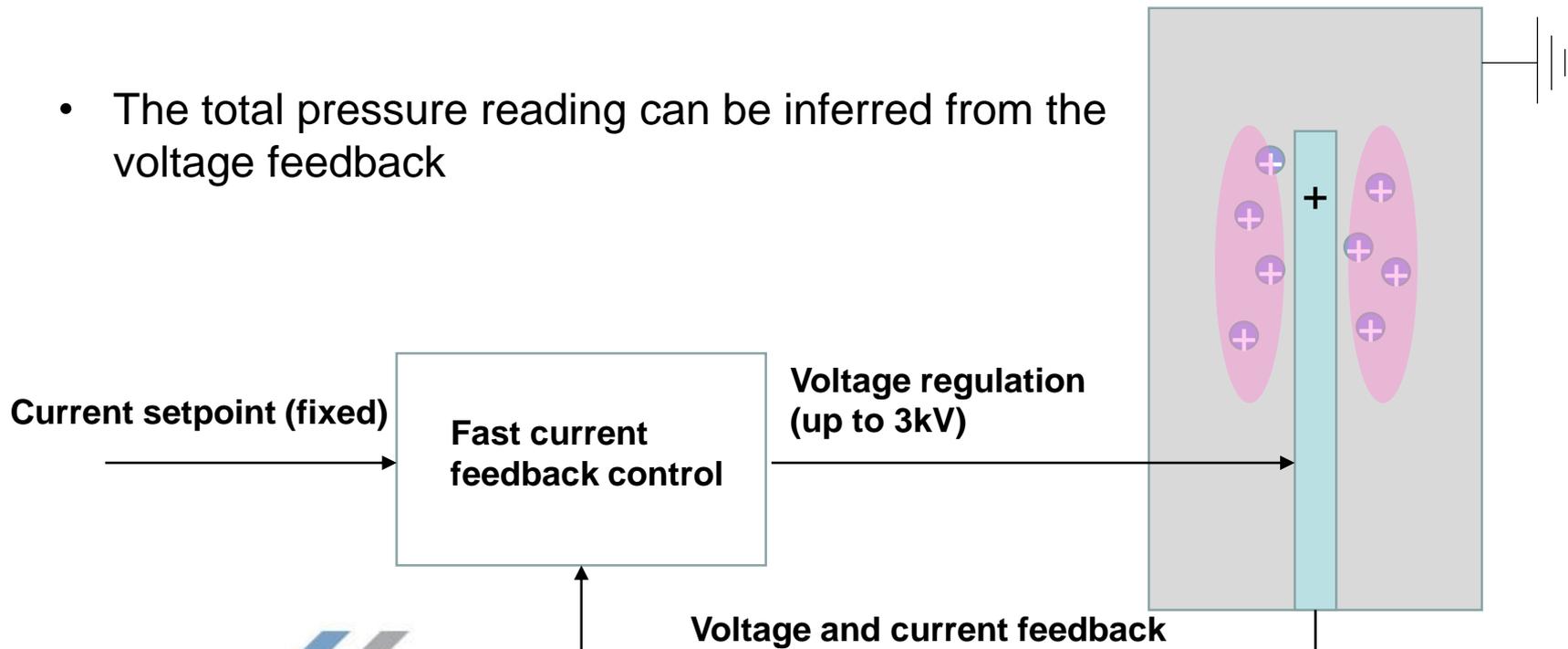
Spectrum analysis gives species composition



Remote plasma generator

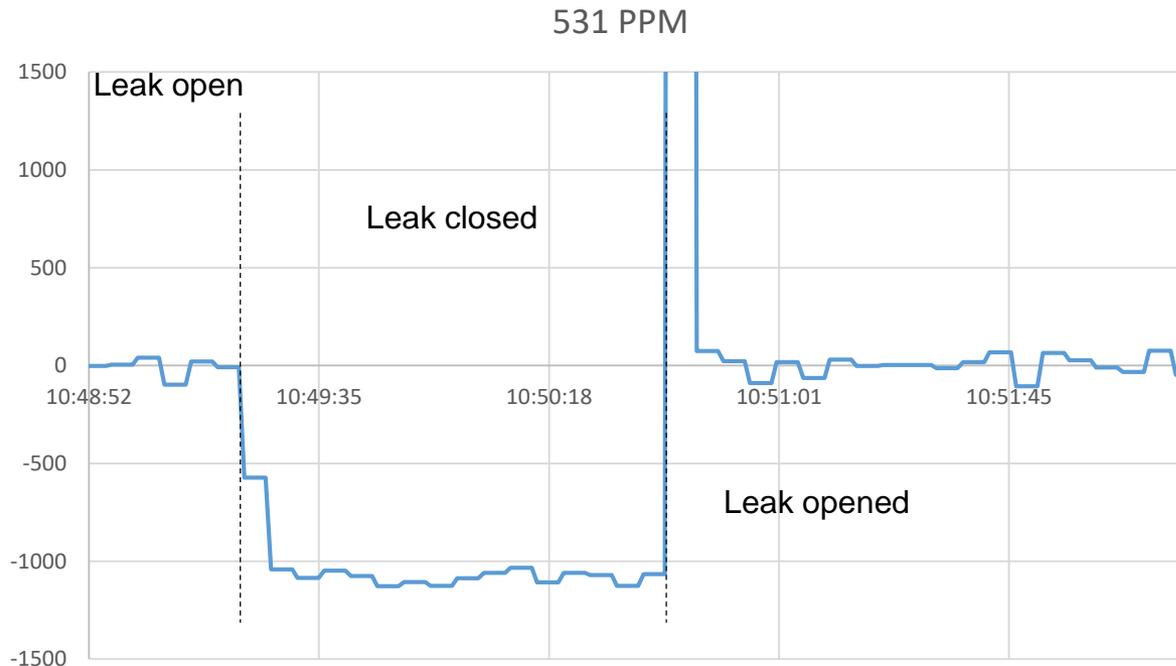


- Fast feedback control of the current allows for a stable plasma to be generated from **1E-6 mbar to 1 mbar**
- Overall species excitation is determined by the current setpoint.
- The total pressure reading can be inferred from the voltage feedback



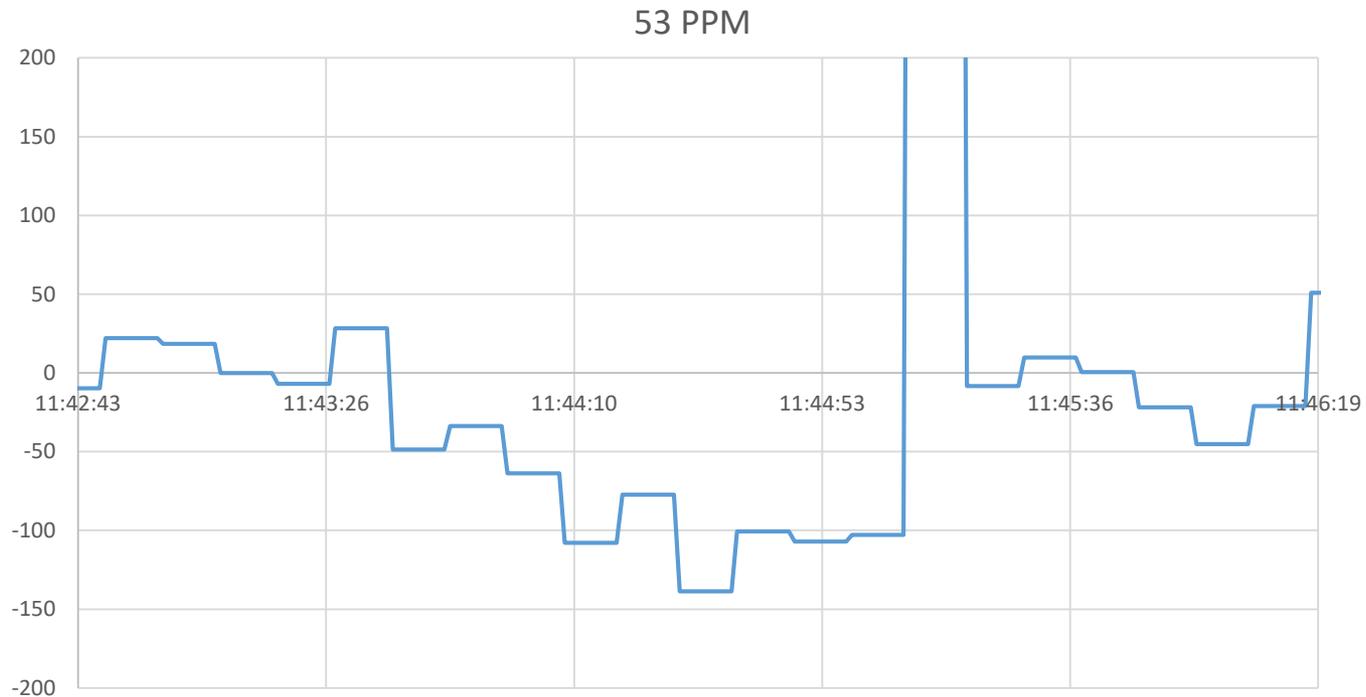
Sensitivity of RPEM

- 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



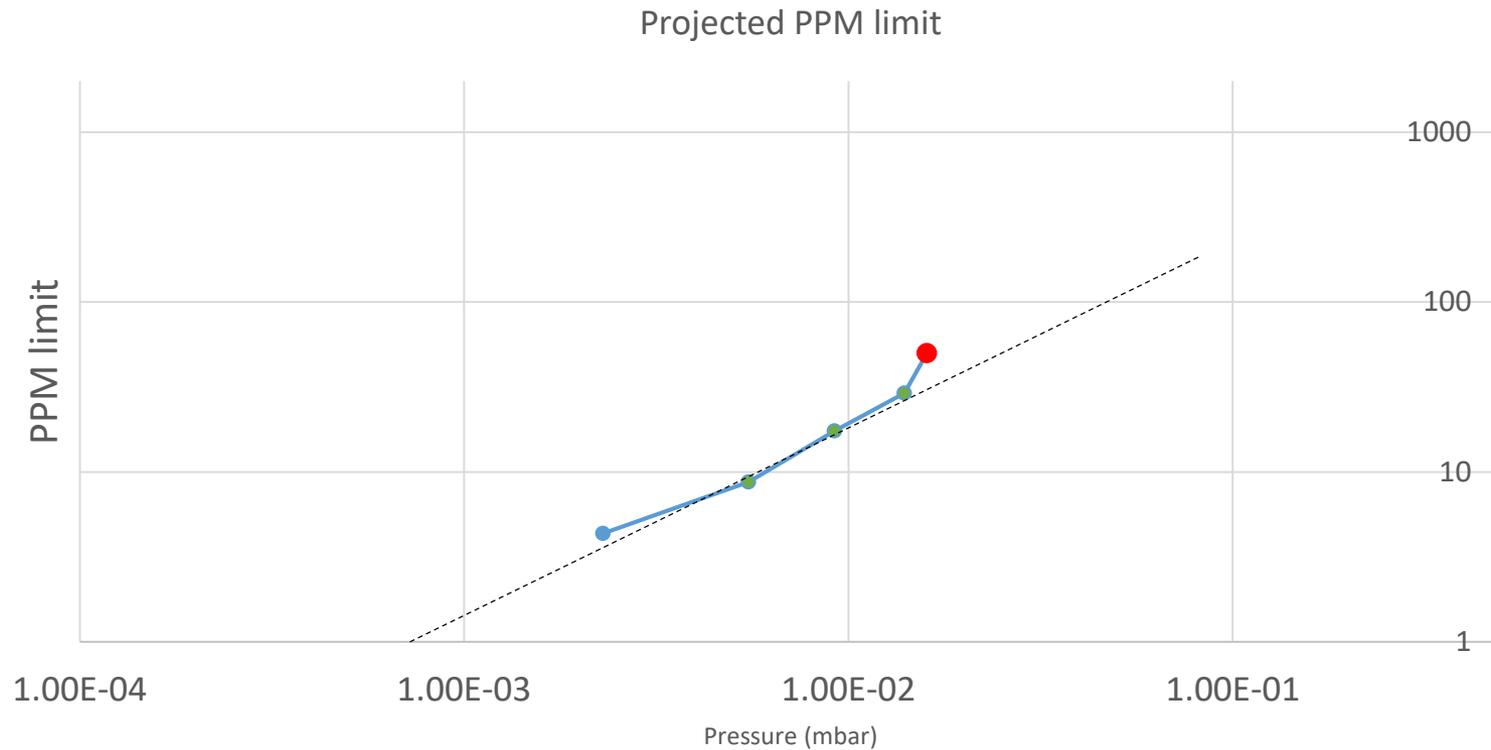
Sensitivity of RPEM

- 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



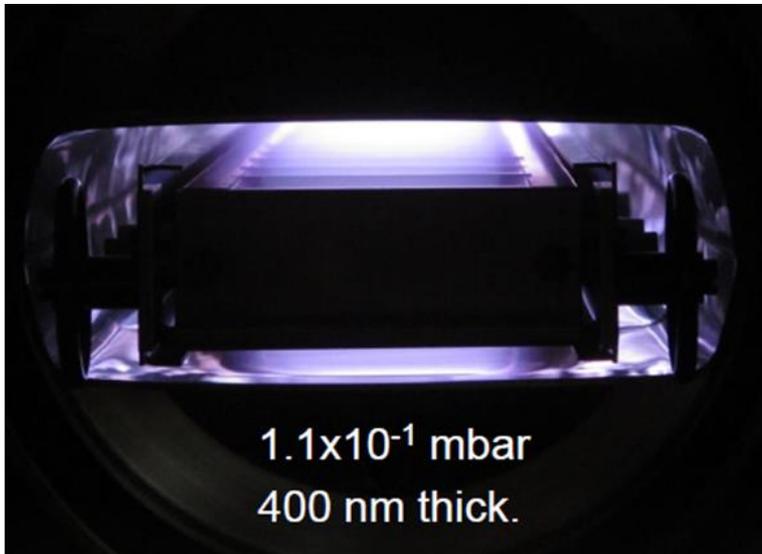
Sensitivity of RPEM

- 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

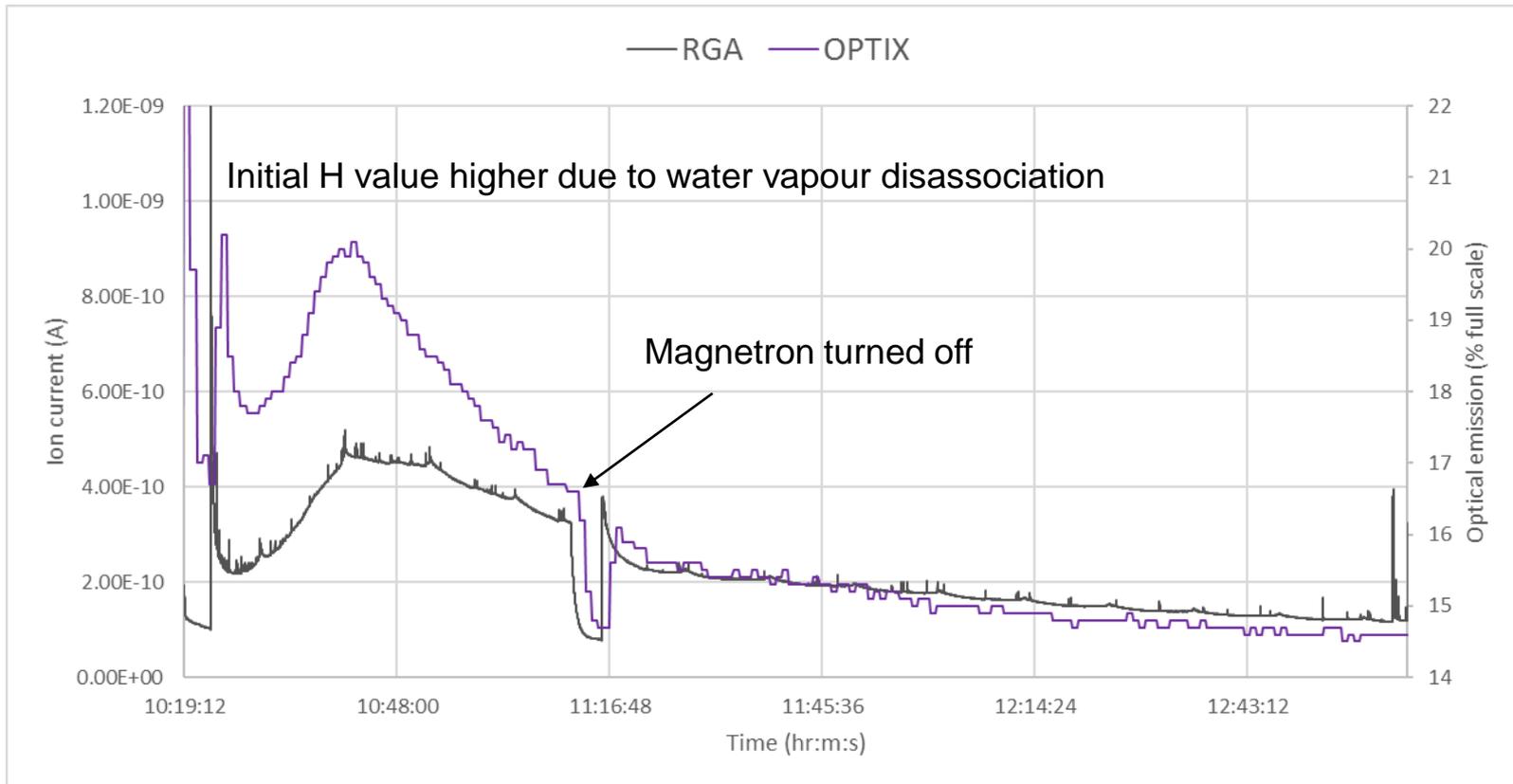
Courtesy of CERN Vacuum Surfaces and Coatings Group



- Carbon sputtered coating
- Deposited on particle accelerator inner surface to reduce secondary electron yield
- Deposition pressure of **1.1E-1 mbar**
- Performance of coating is sensitive to the presence of H outgassing from the magnetron
- Objective to monitor H outgassing during the deposition

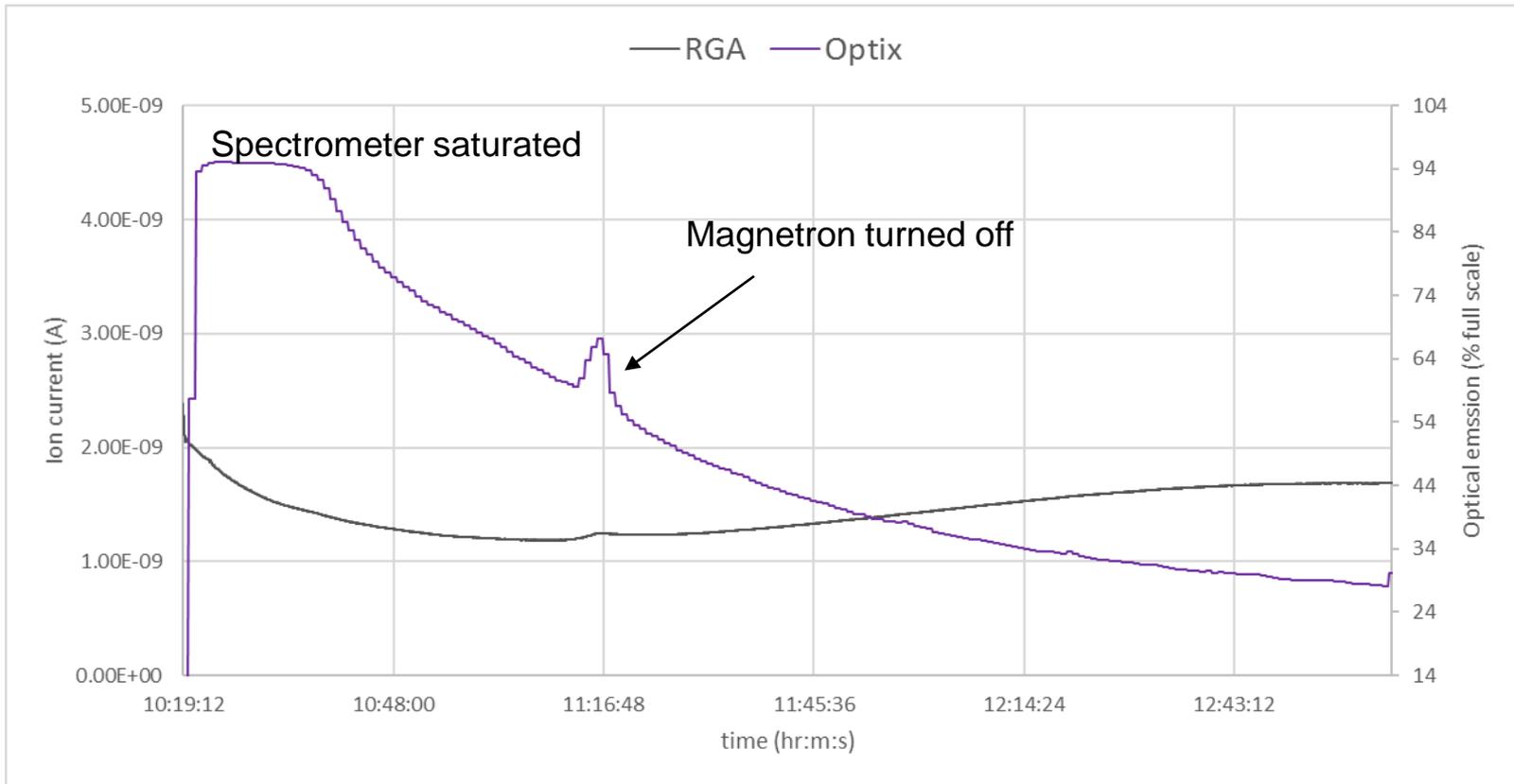
Case study 1 – Outgassing measurement during carbon coating process

Hydrogen - 656 nm, 2 AMU



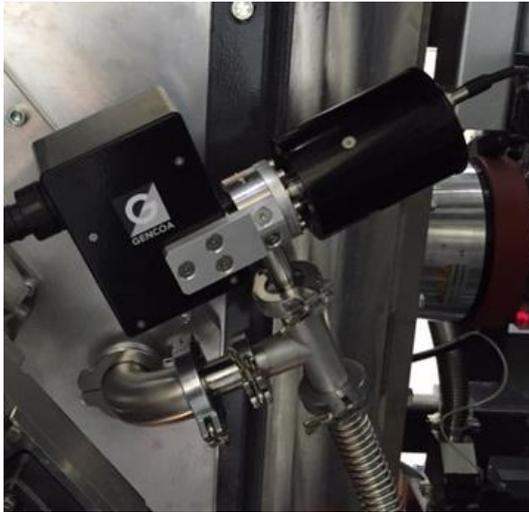
Case study 1 – Outgassing measurement during carbon coating process

Water vapour - 309.6 nm (OH), 18 AMU



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

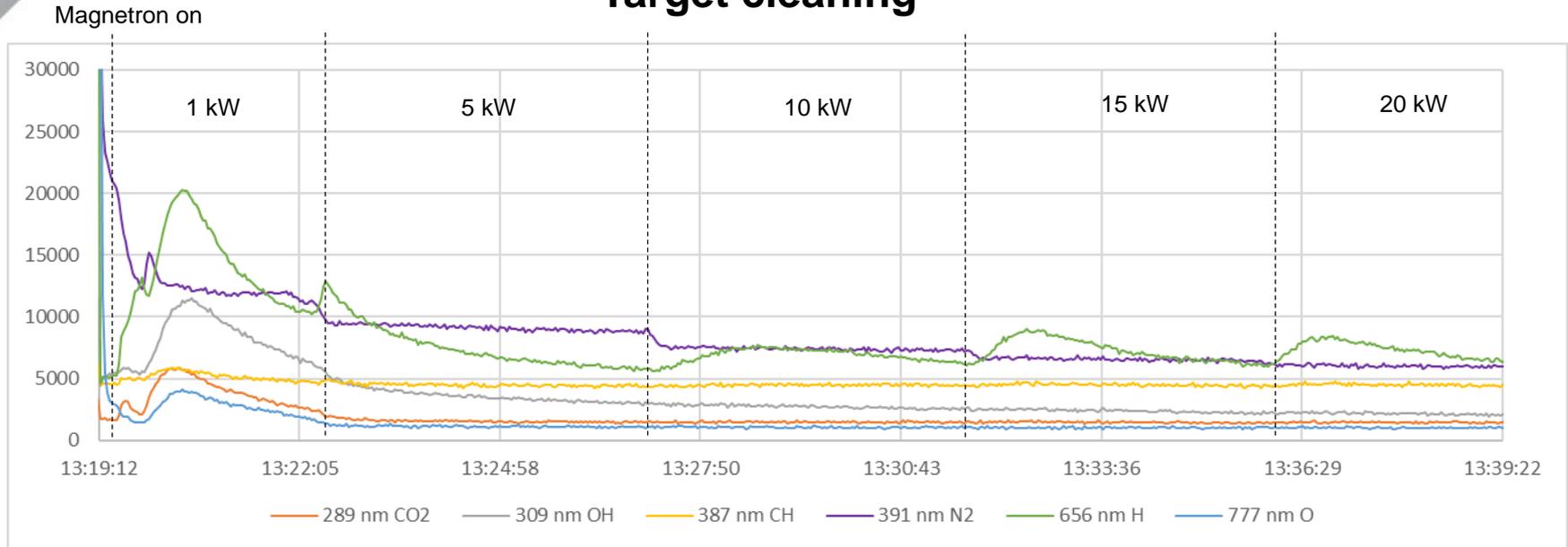
- Roll-to-roll deposition of reactively sputtered AlOx onto 125µm PET
- Optix sensor teed with a differentially pumped RGA



Courtesy of Emerson and Renwick

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

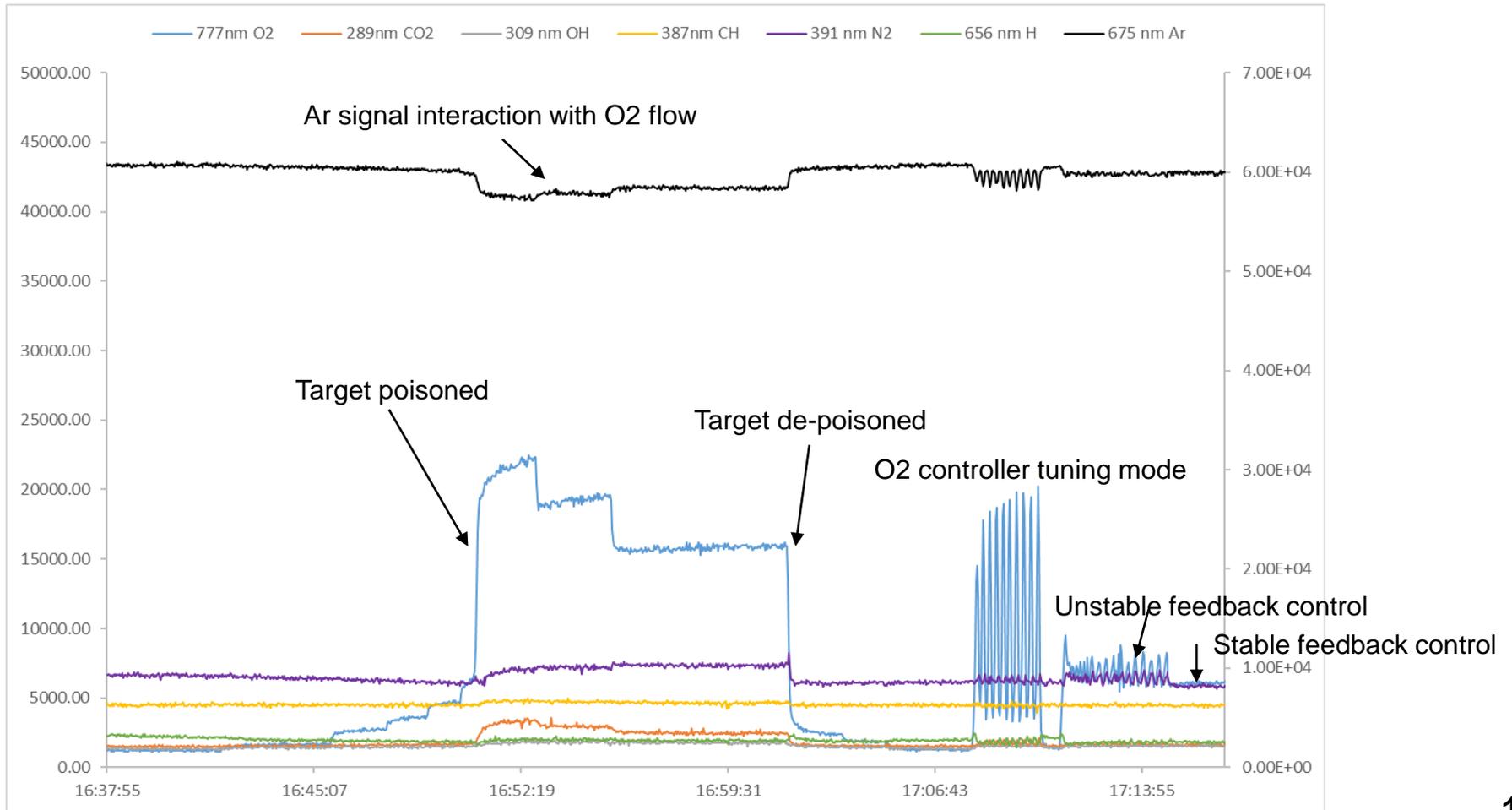
Target cleaning



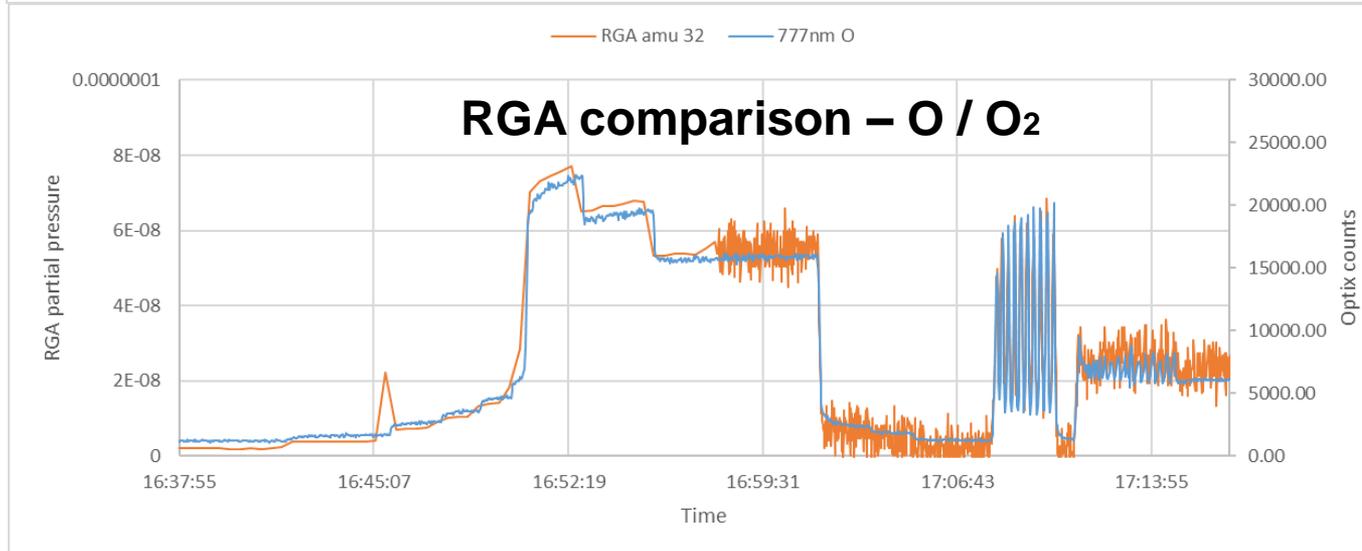
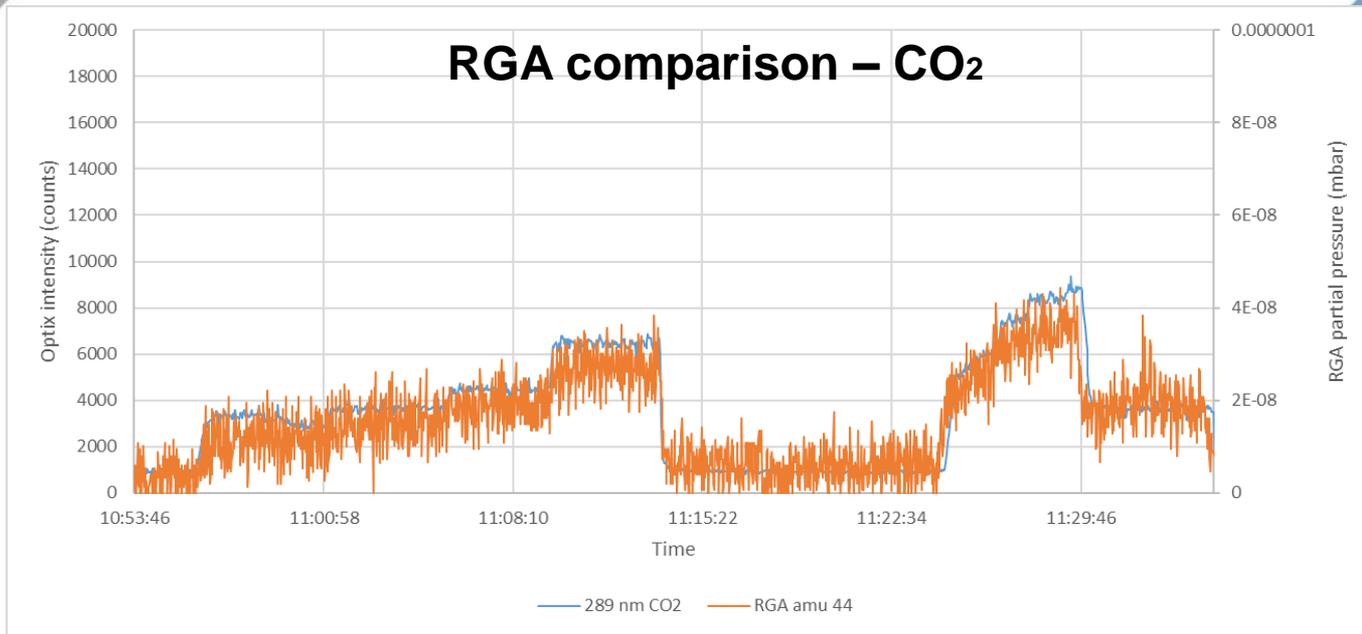
- Very large H outgassing – taking significant time to reach steady state
- Other species also observed initially outgassing – OH, CO2, O
- Subsequent power increases cause increased H outgassing and additional settling time
- Consumption of N2 also observed – small chamber leak

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

Reactive sputter characterisation

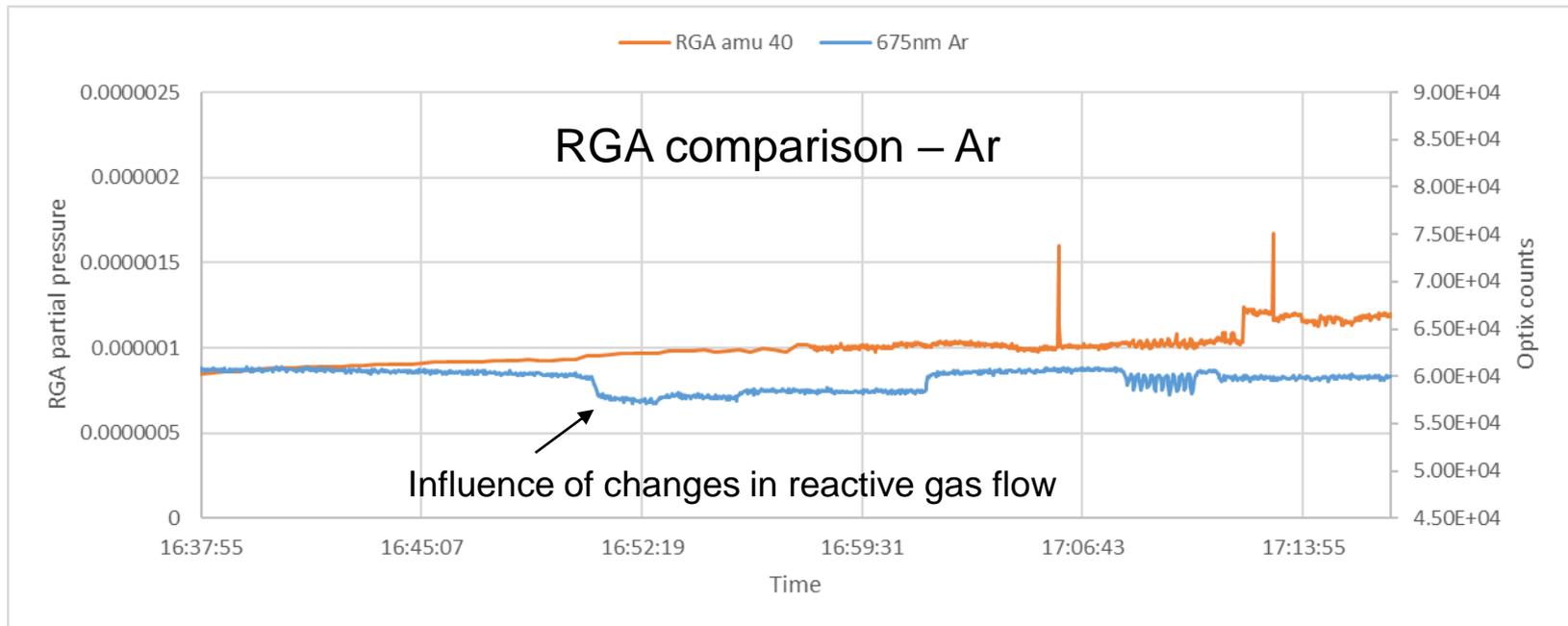


Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

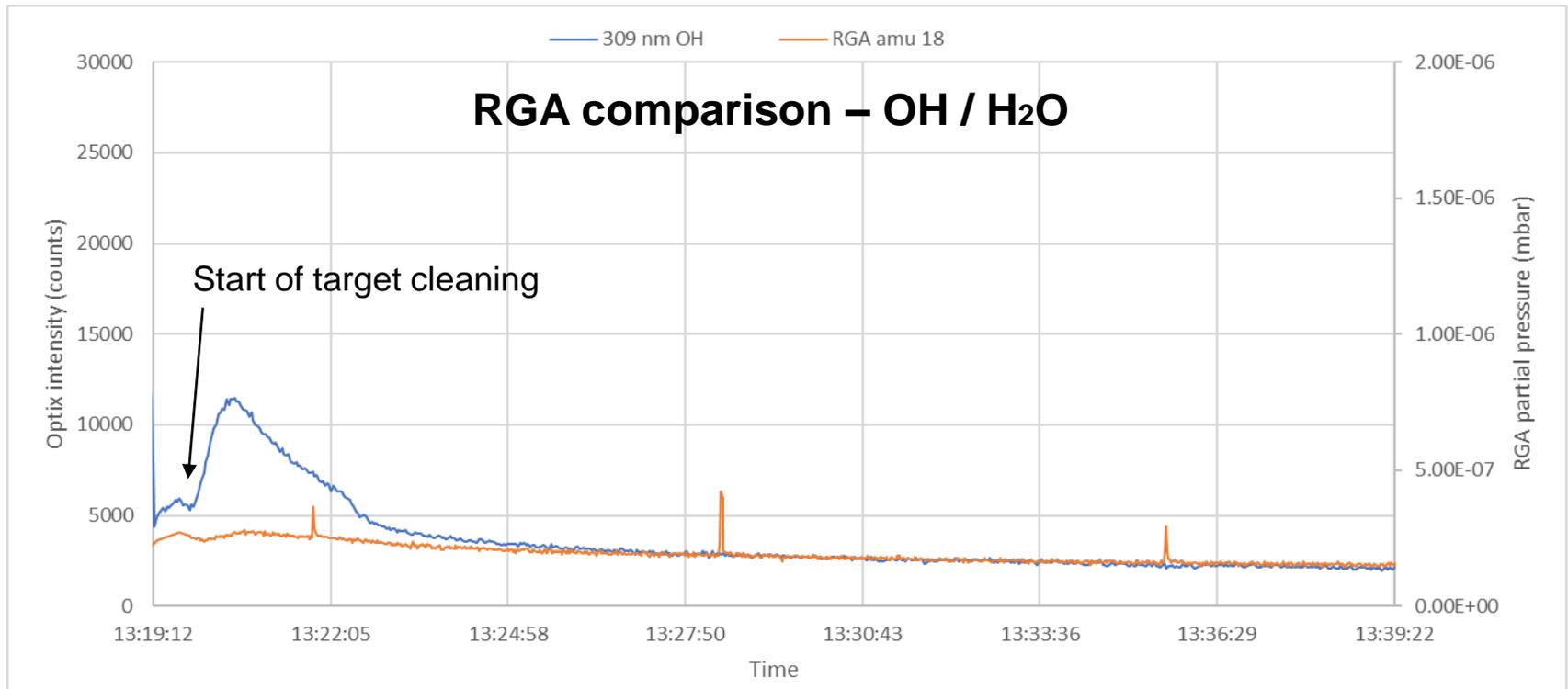


Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

- 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

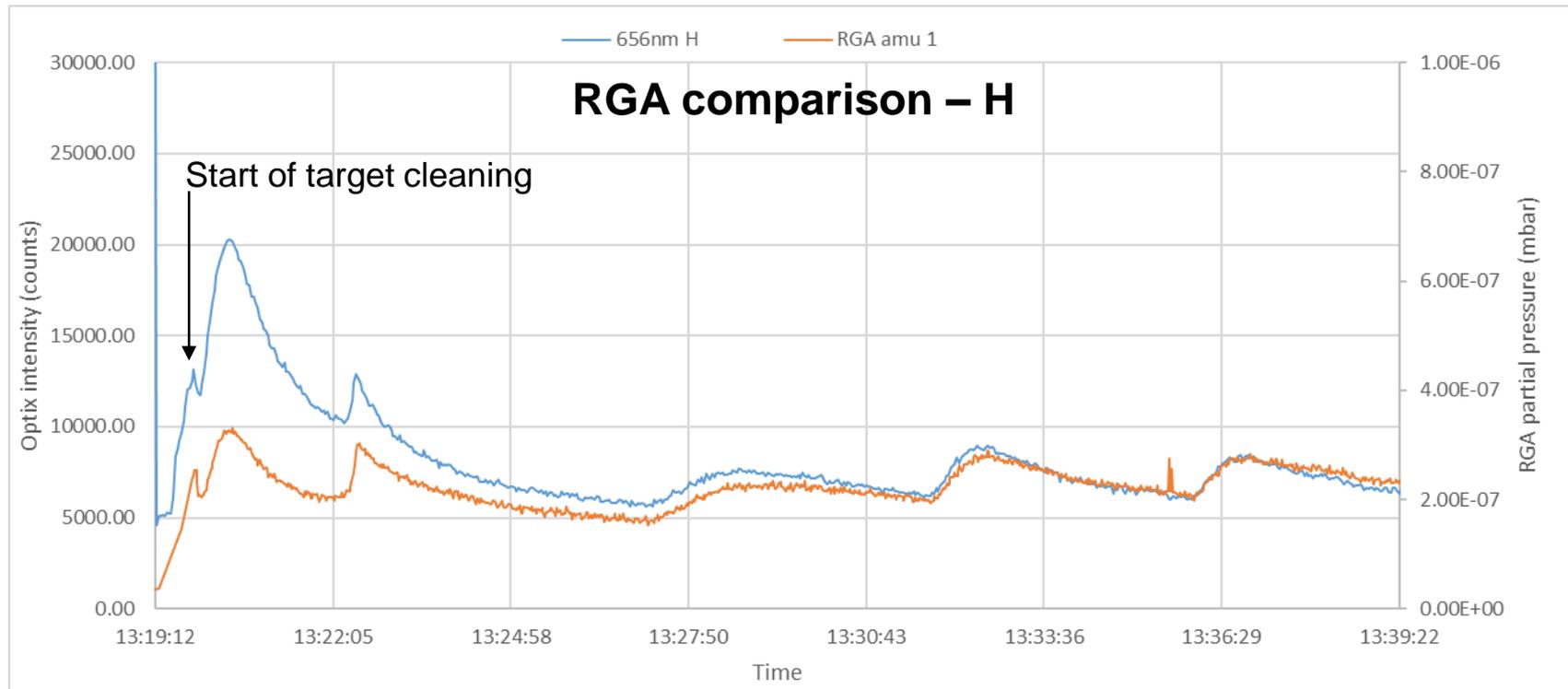


Case study 2 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web



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.

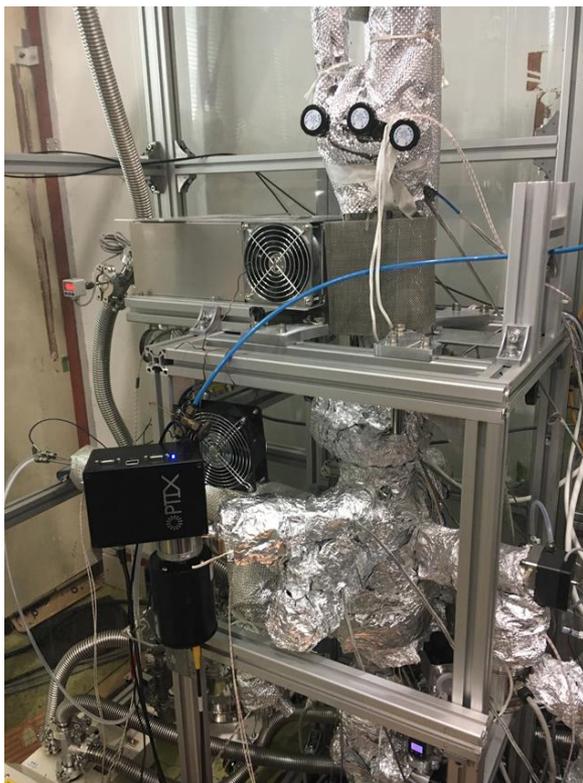


Case study 3 – Atomic Layer Deposition

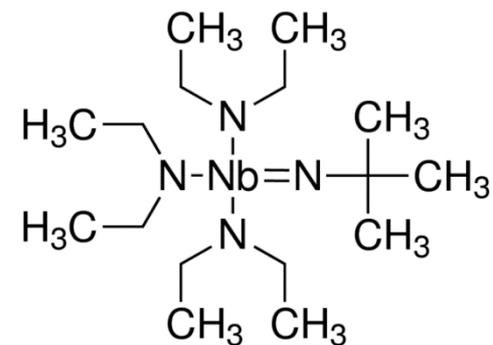
Deposition of NbN via PEALD

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

Image and data courtesy of S. Kato



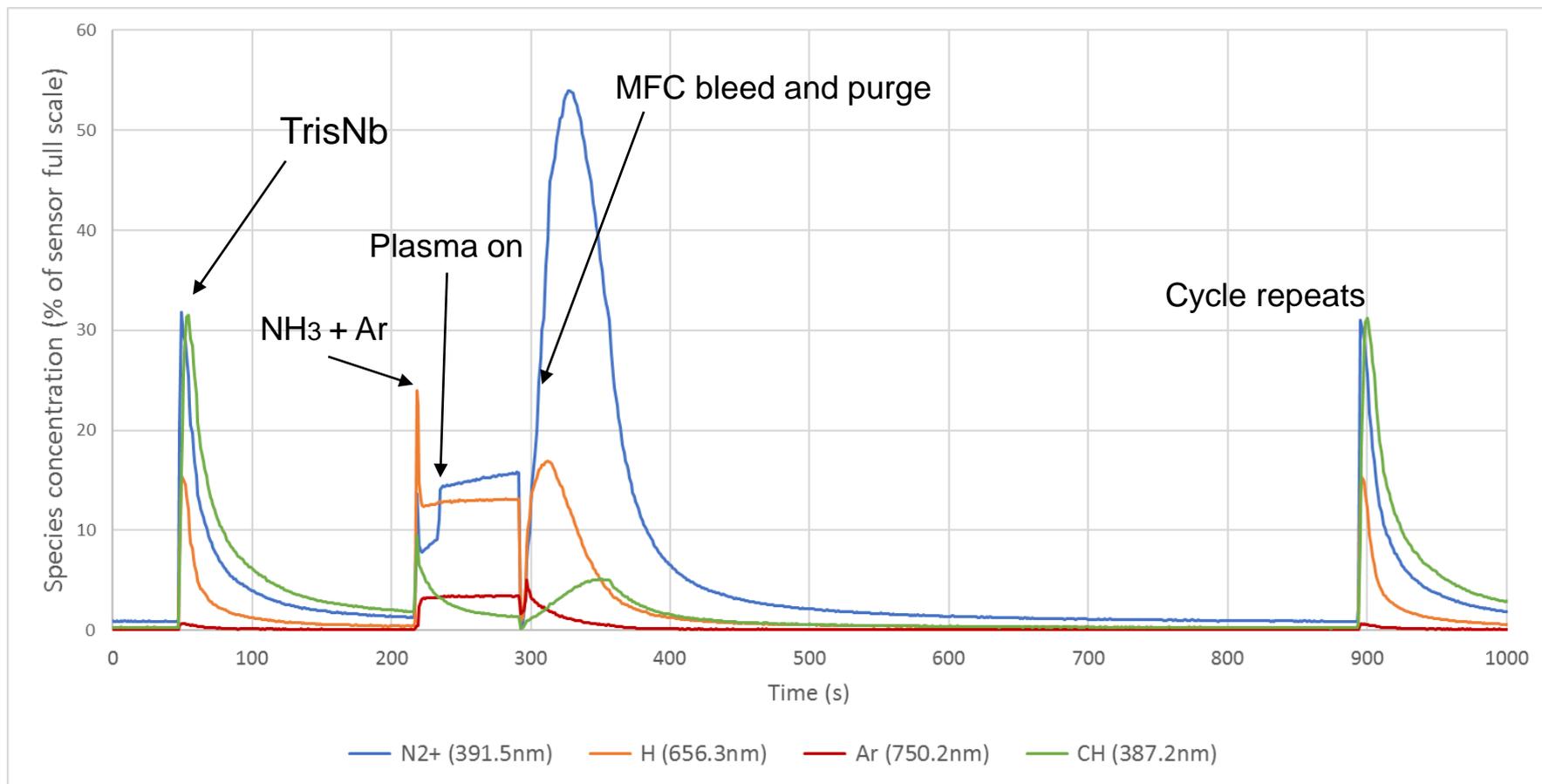
- Detection of TrisNb via CH, N and H



- Detection of NH₃ via N and H

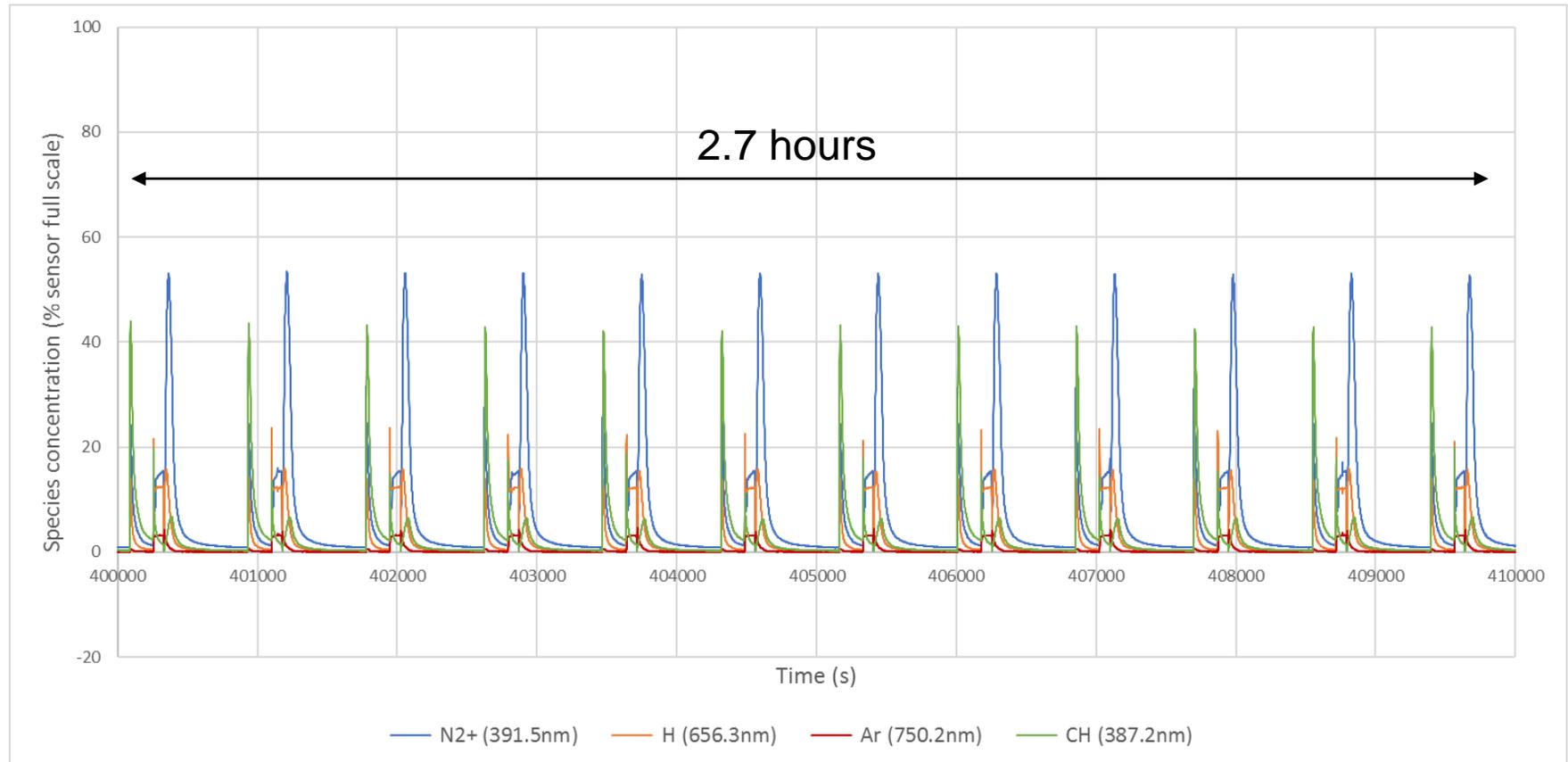
Case study 3 – Atomic Layer Deposition

NbN deposition cycle



Case study 3 – Atomic Layer Deposition

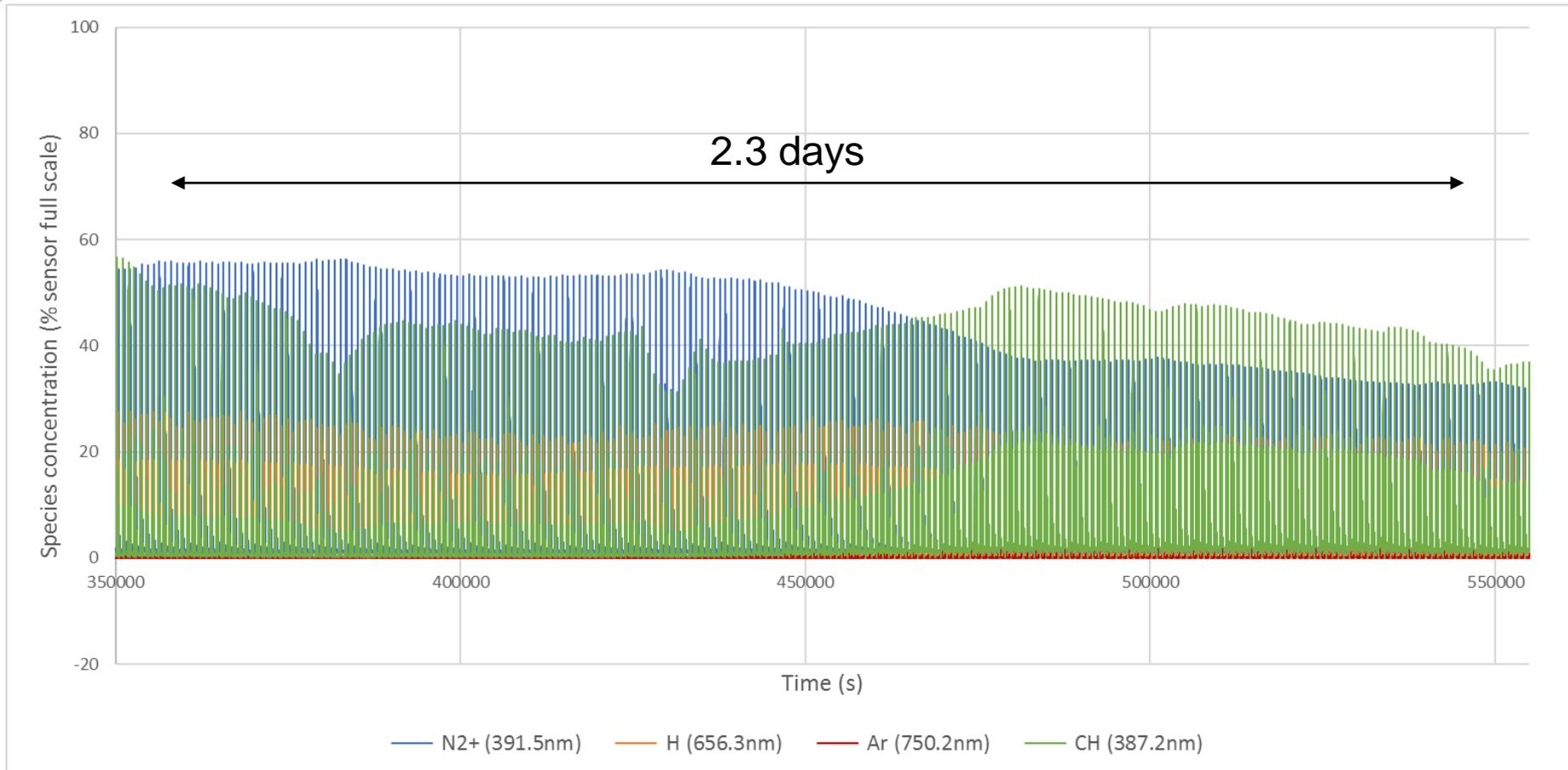
NbN deposition cycle



Case study 3 – Atomic Layer Deposition

NbN deposition cycle

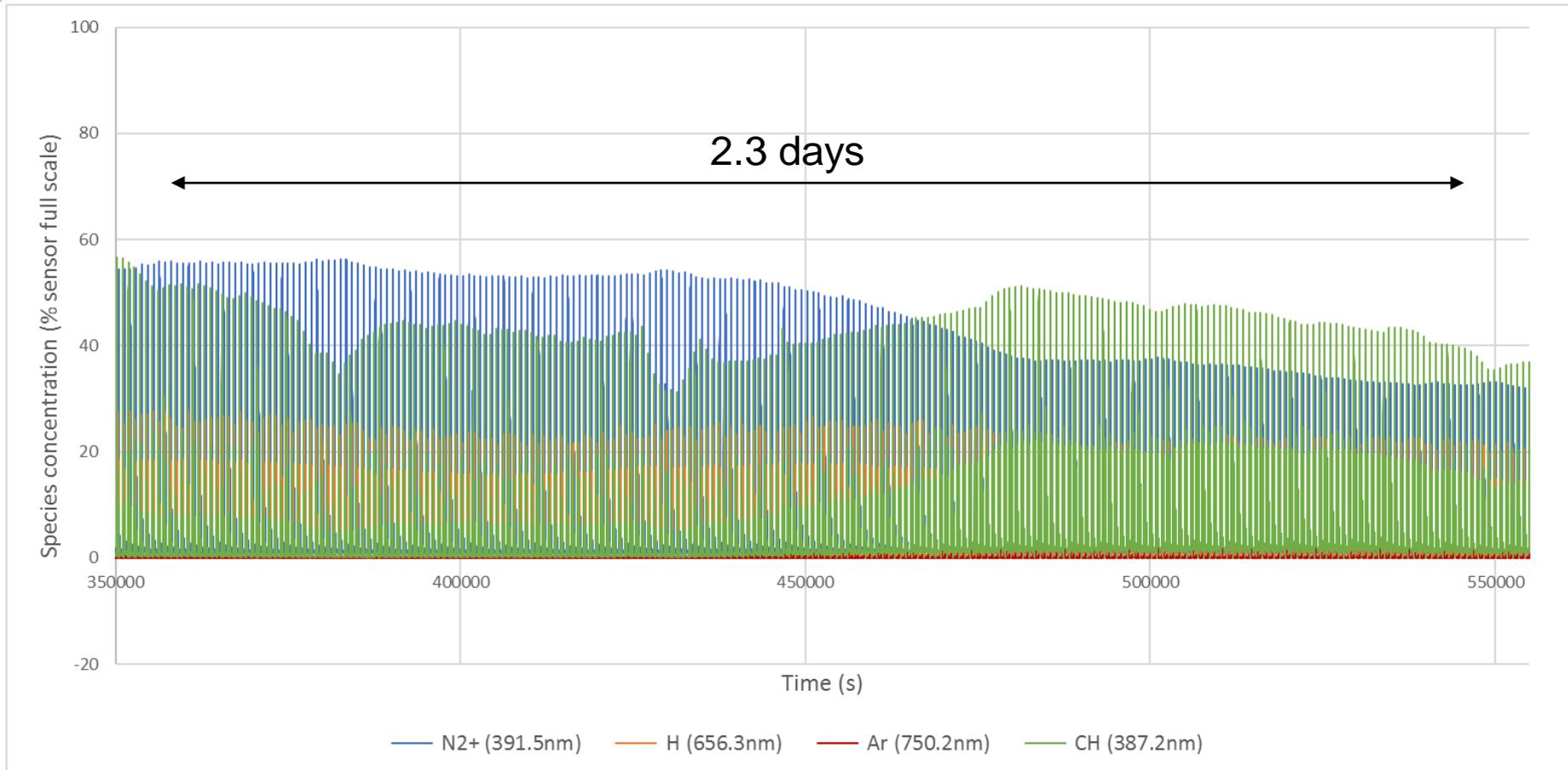
- Sensor is robust of the full 2+ day deposition cycle



Case study 3 – Atomic Layer Deposition

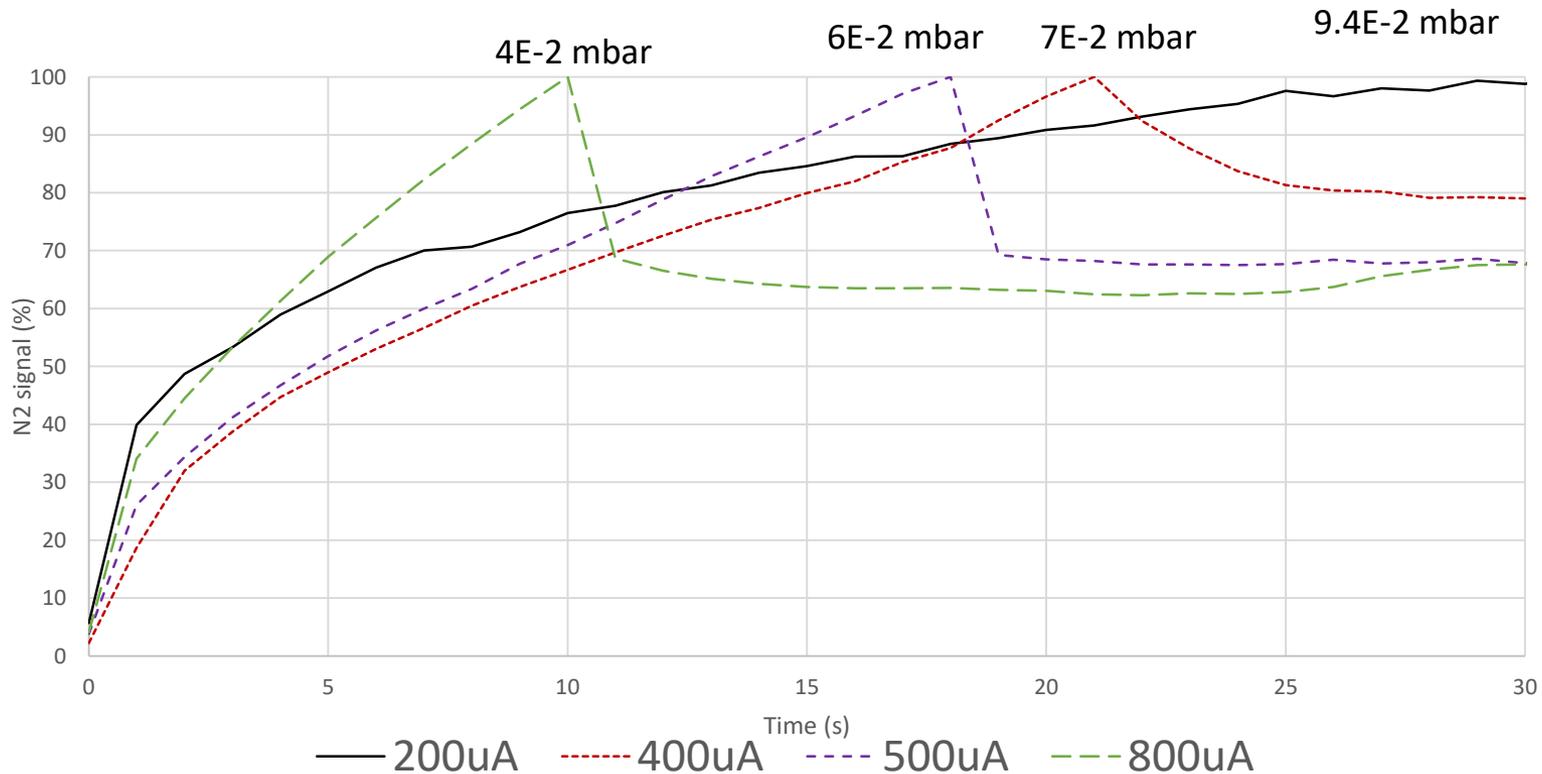
NbN deposition cycle

- Sensor is robust of the full 2+ day deposition cycle



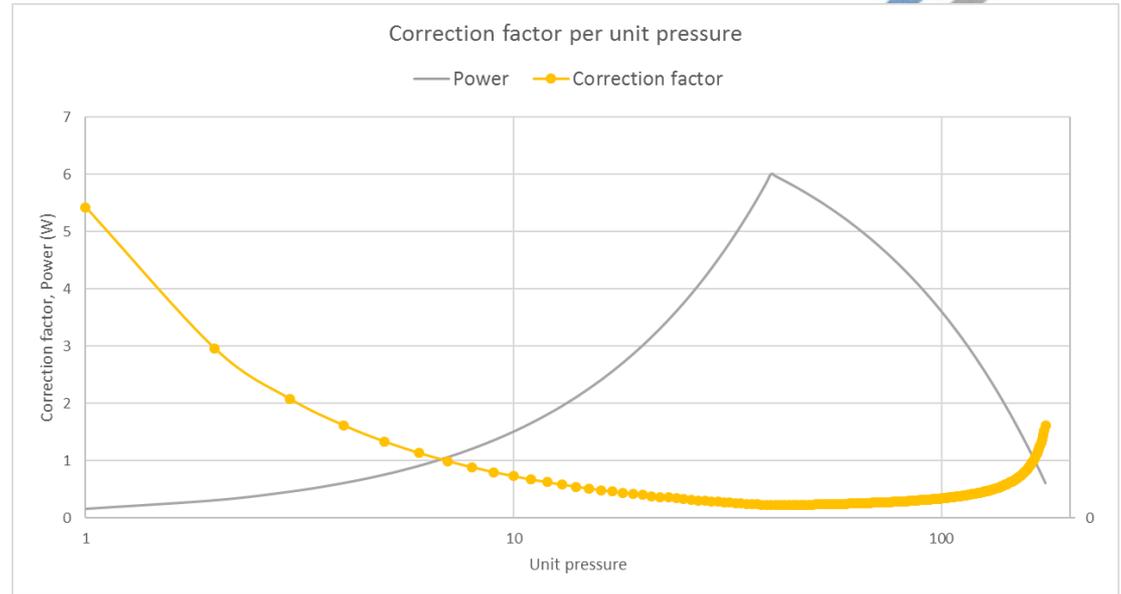
Quantification - Pressure limitations

- 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

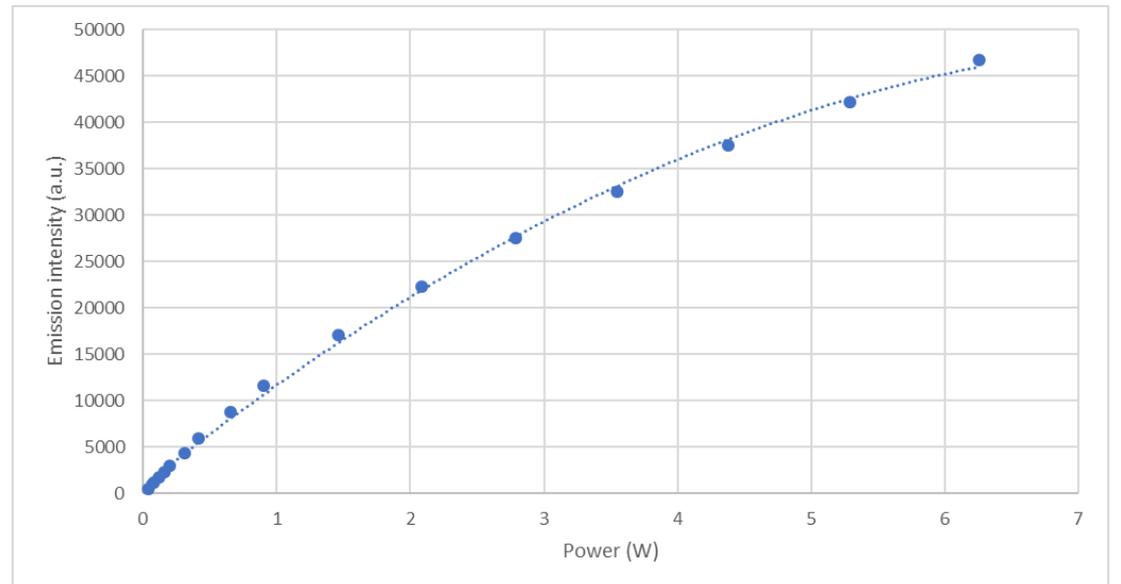


Quantification - Power correction

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

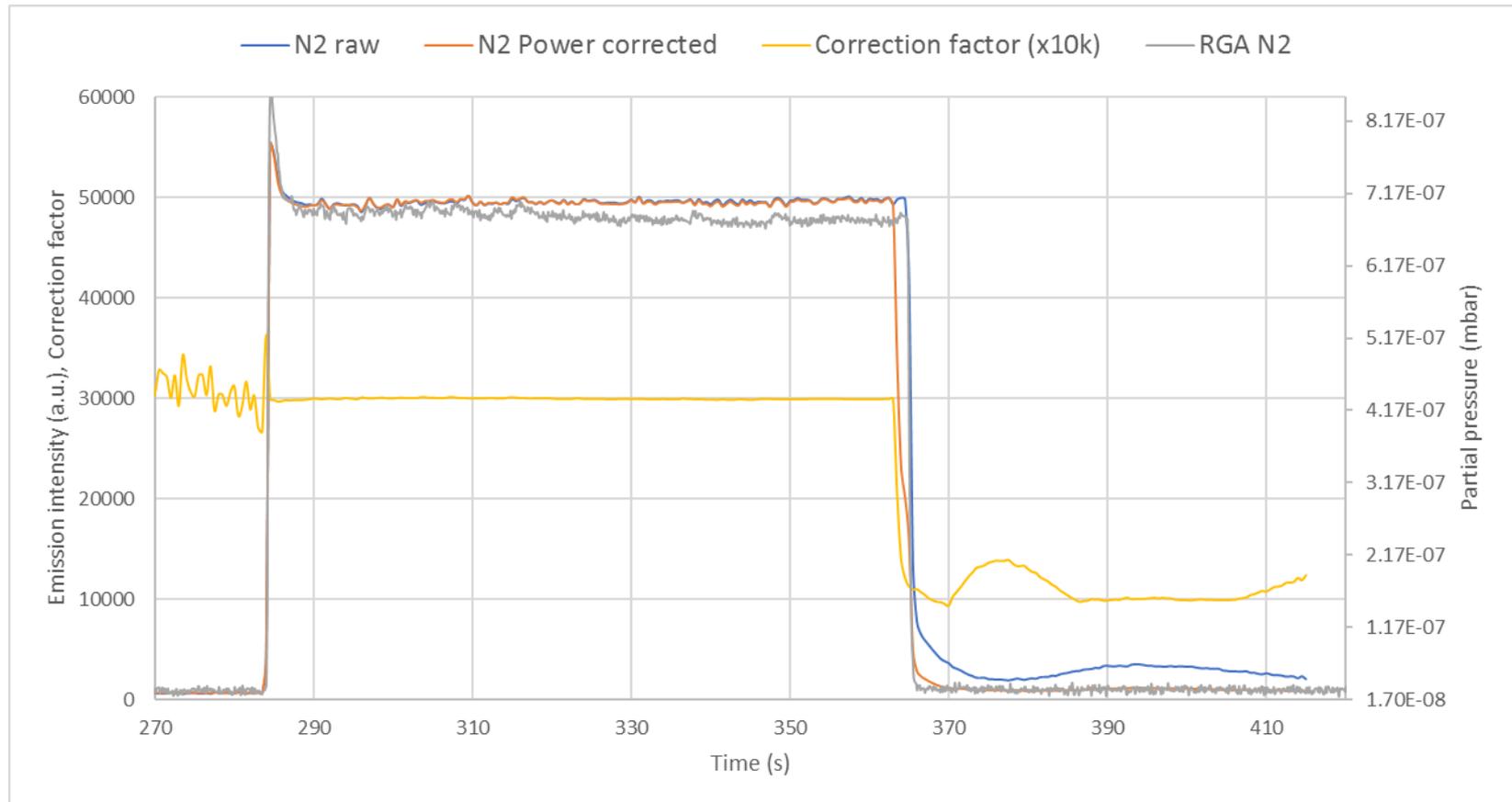


- The power delivered to the plasma generator will modify the emission intensities



Quantification - Power correction

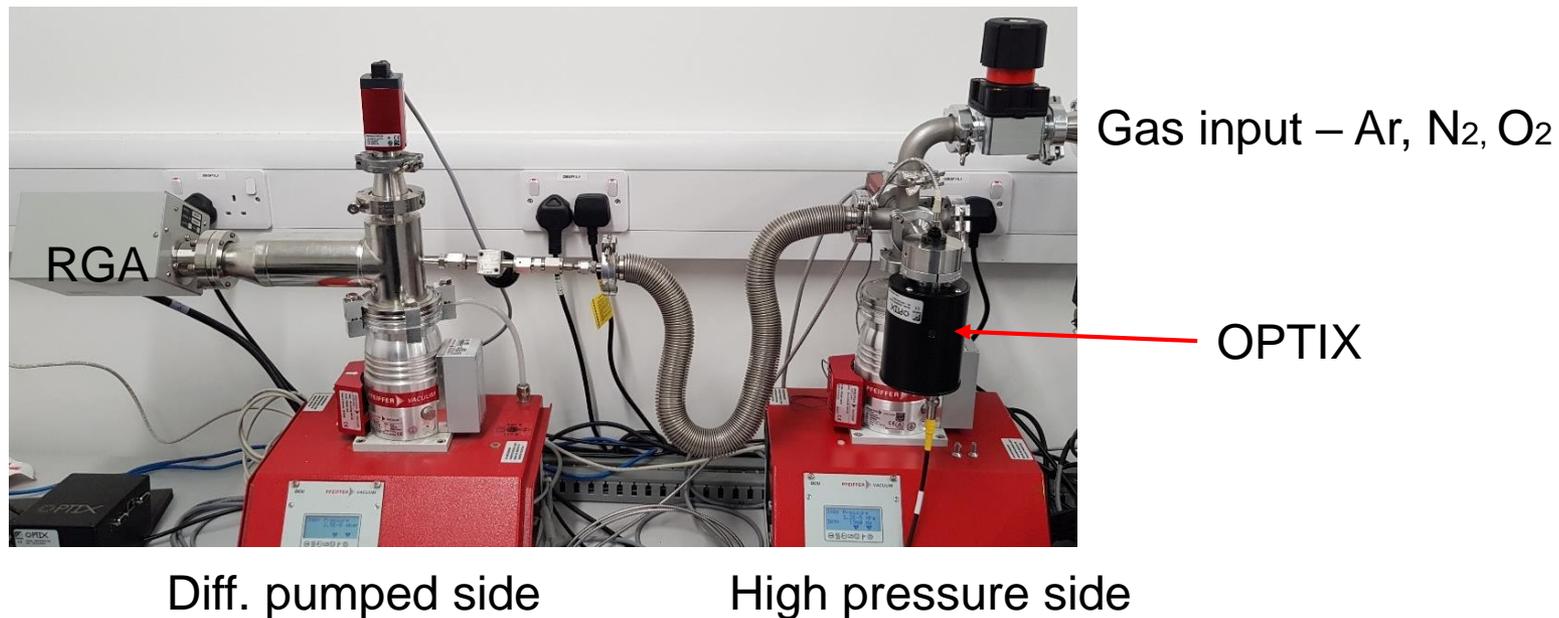
- The effect of the correction can be clearly seen when compared with a differentially pumped RGA



Quantification – Gas interaction

Experimental setup

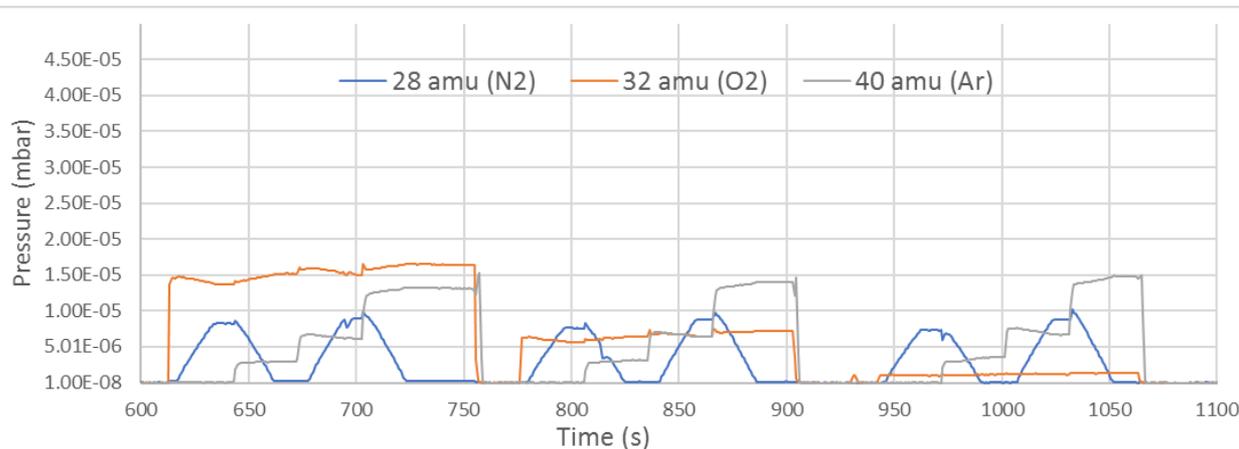
- The most significant challenge for quantification of the sensor readings is the interactivity of gases
- Without correction the readings are **relative** not absolute
- i.e. increasing partial pressure of one gas will lead to a reduction in the readings of other gases.
- An experimental setup was constructed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



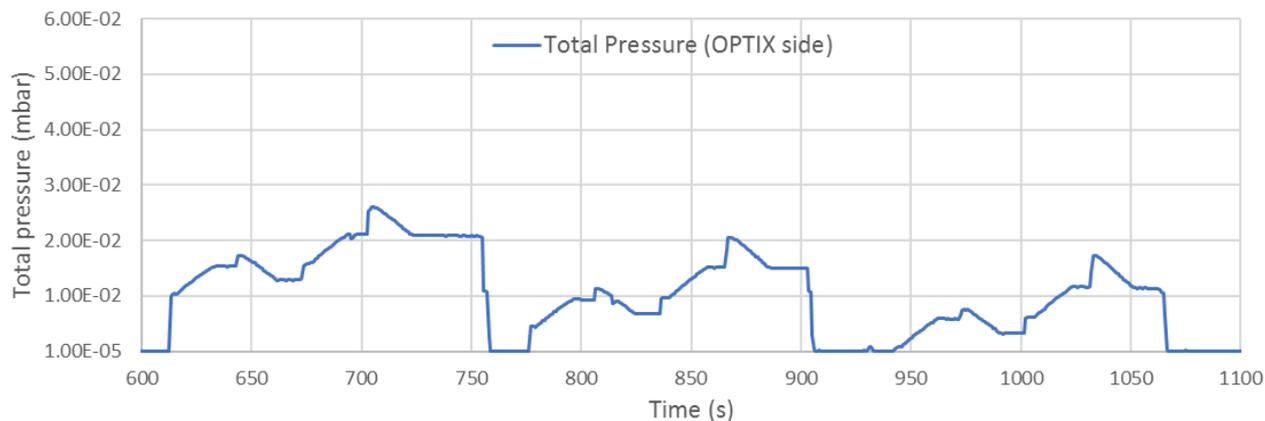
Quantification – Gas interaction

- 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

Diff. pumped RGA



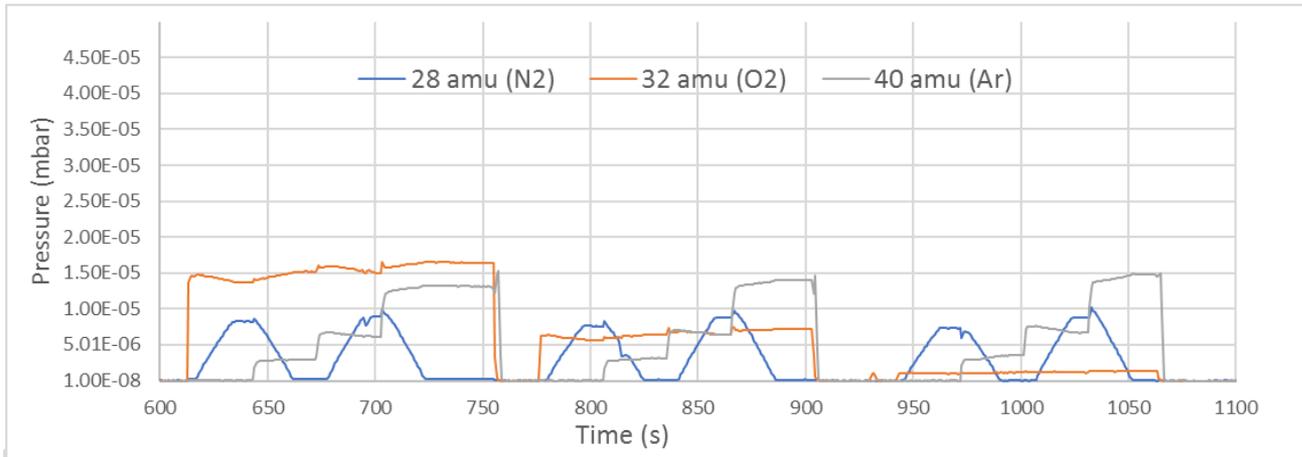
Total pressure



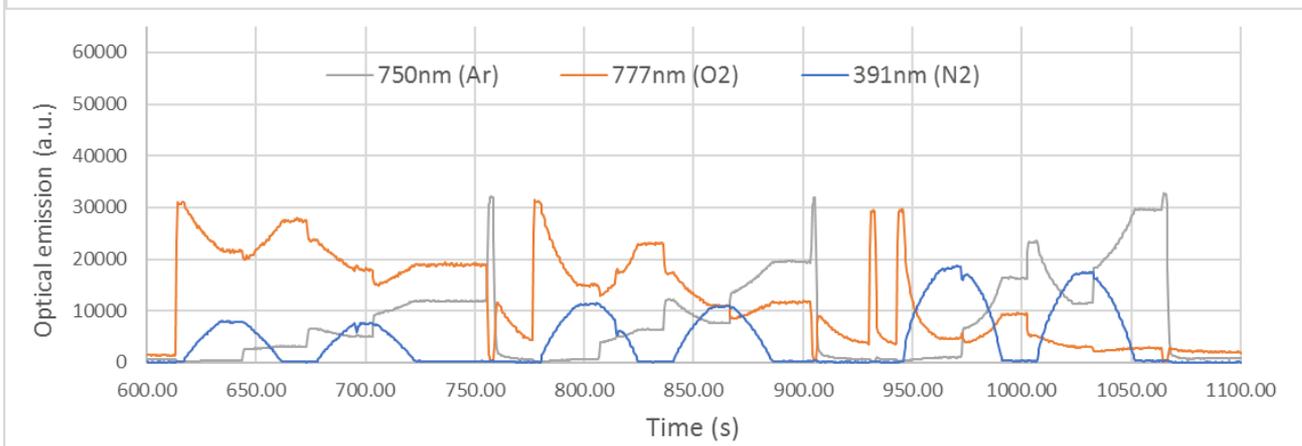
Quantification – Gas interaction

- Gas interaction effects can be clearly seen on the OPTIX readings

Diff. pumped RGA



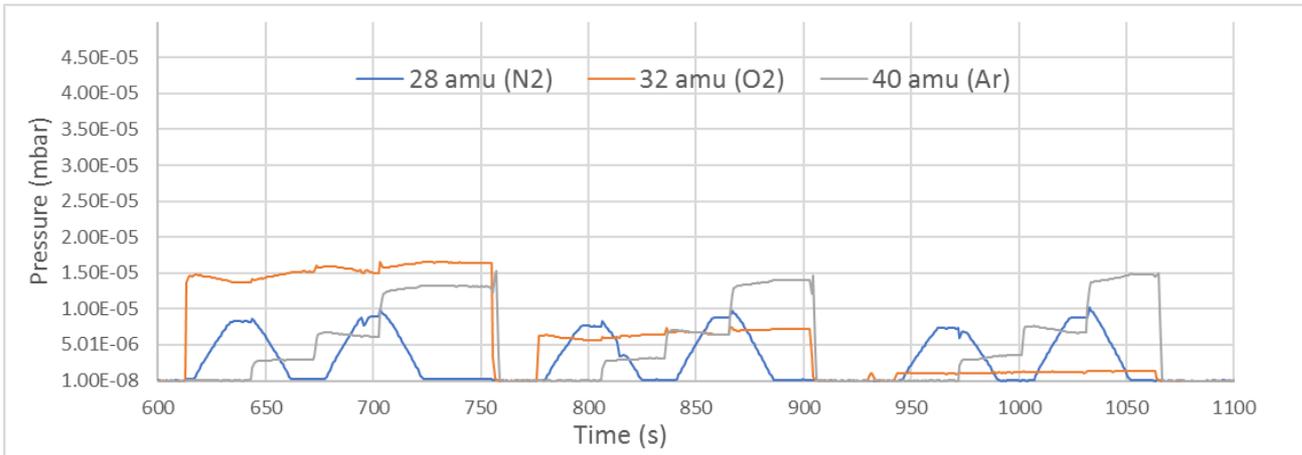
OPTIX readings



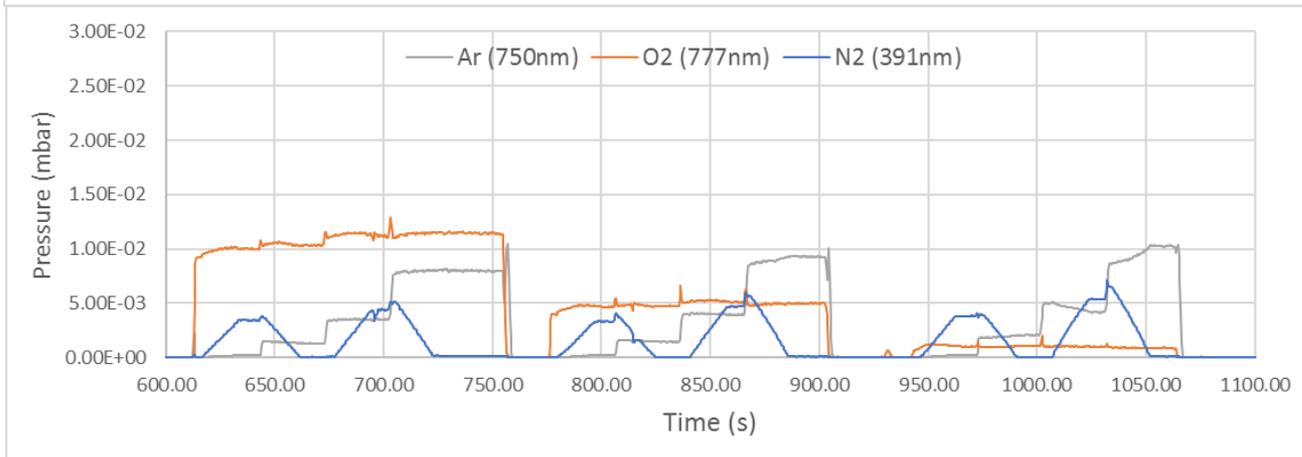
Quantification – Gas interaction

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

Diff. pumped RGA

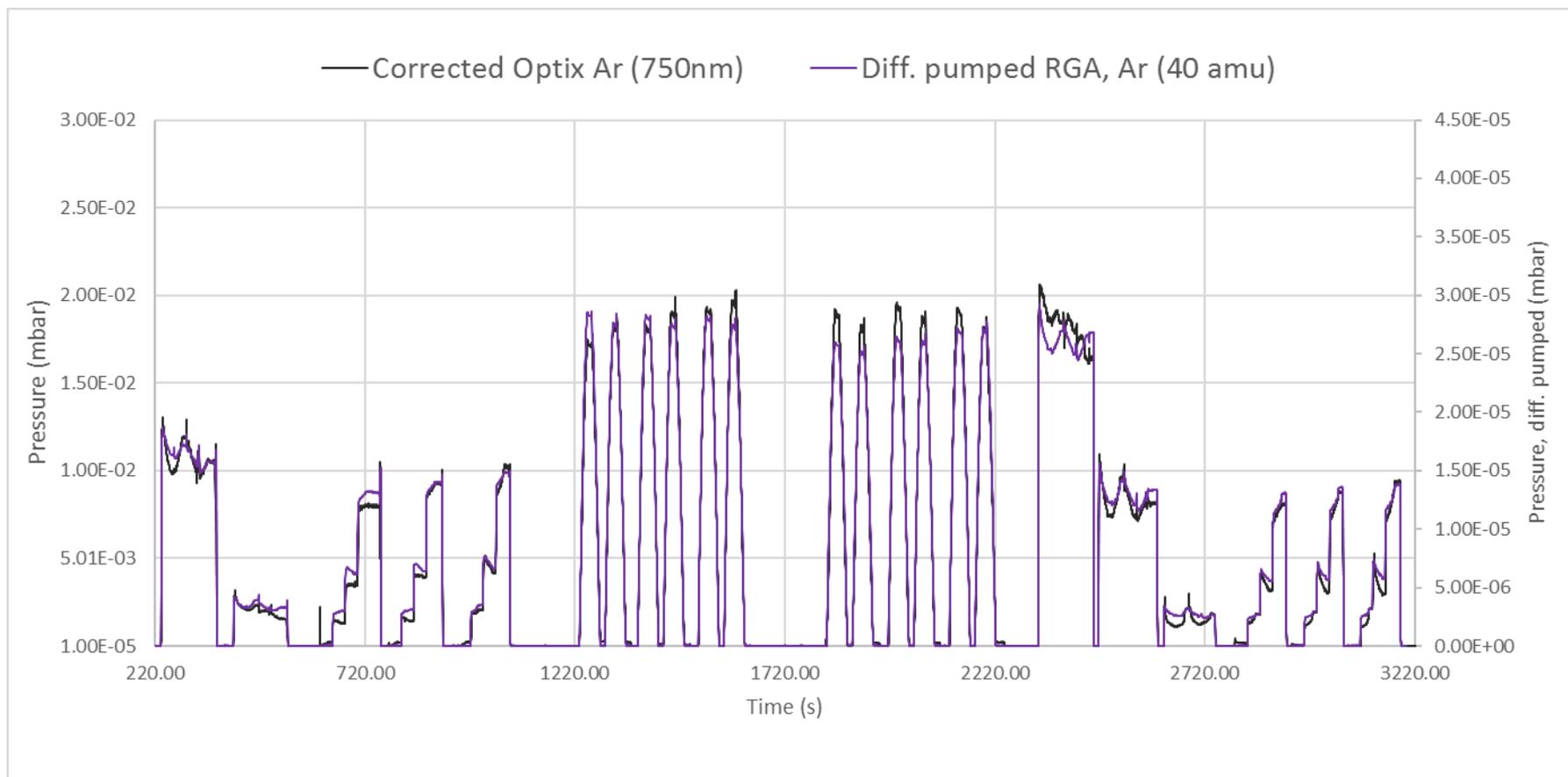


OPTIX readings



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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