



AIMCAL R2R 2017
Oct 15th – 18th 2017

Monitoring and control of web coating sputtering processes using remote optical spectroscopy gas sensing

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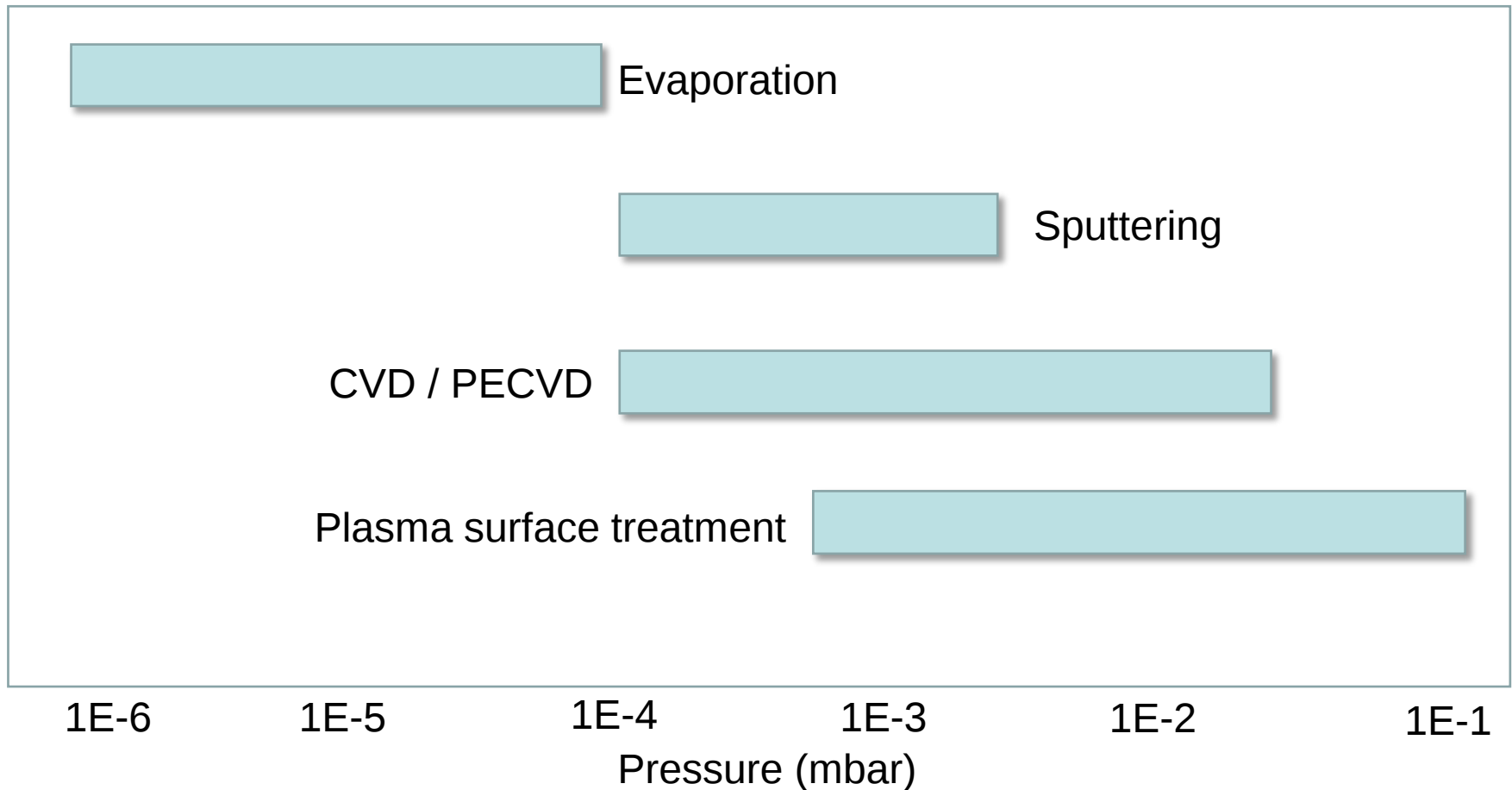
Gencoia Limited, Liverpool, UK

*** Gencoia USA**

- Description of the sensing technique
- Monitoring of an ALD process
- Reactive sputter monitoring and comparisons with RGA data
- Atmospheric vapour detection
- Obtaining quantitative measurements

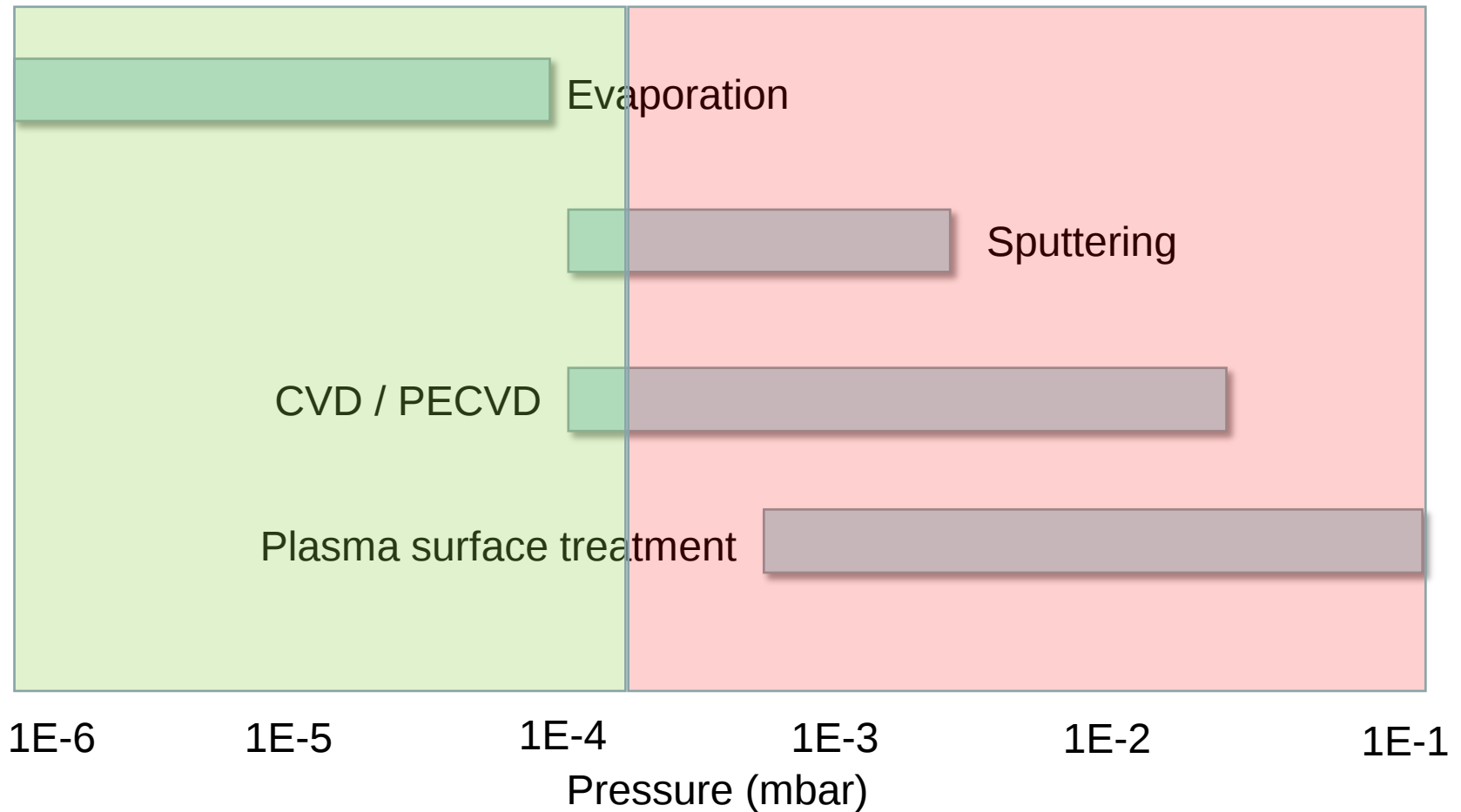
Motivation for the sensing technique

Typical vacuum deposition and surface treatment pressure ranges



Motivation for the sensing technique

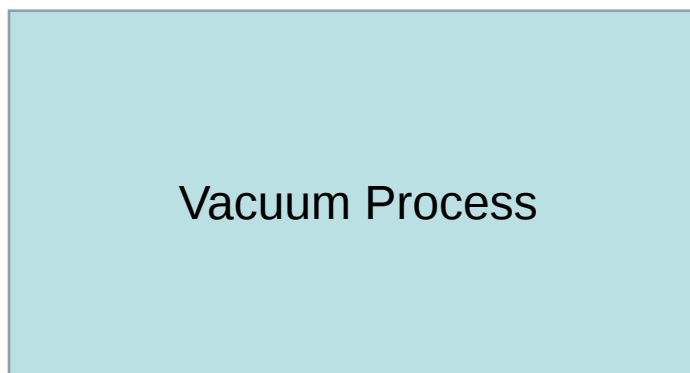
Typical vacuum deposition and surface treatment pressure ranges



Overview of the sensing technique

Remote Plasma Emission Spectroscopy

Spectrum analysis
gives species composition



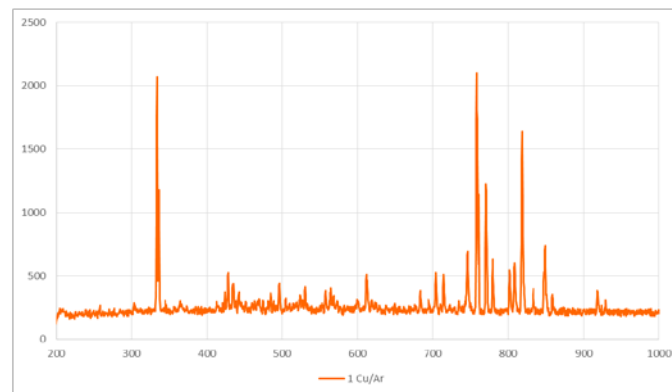
Remote
plasma
generator



Plasma

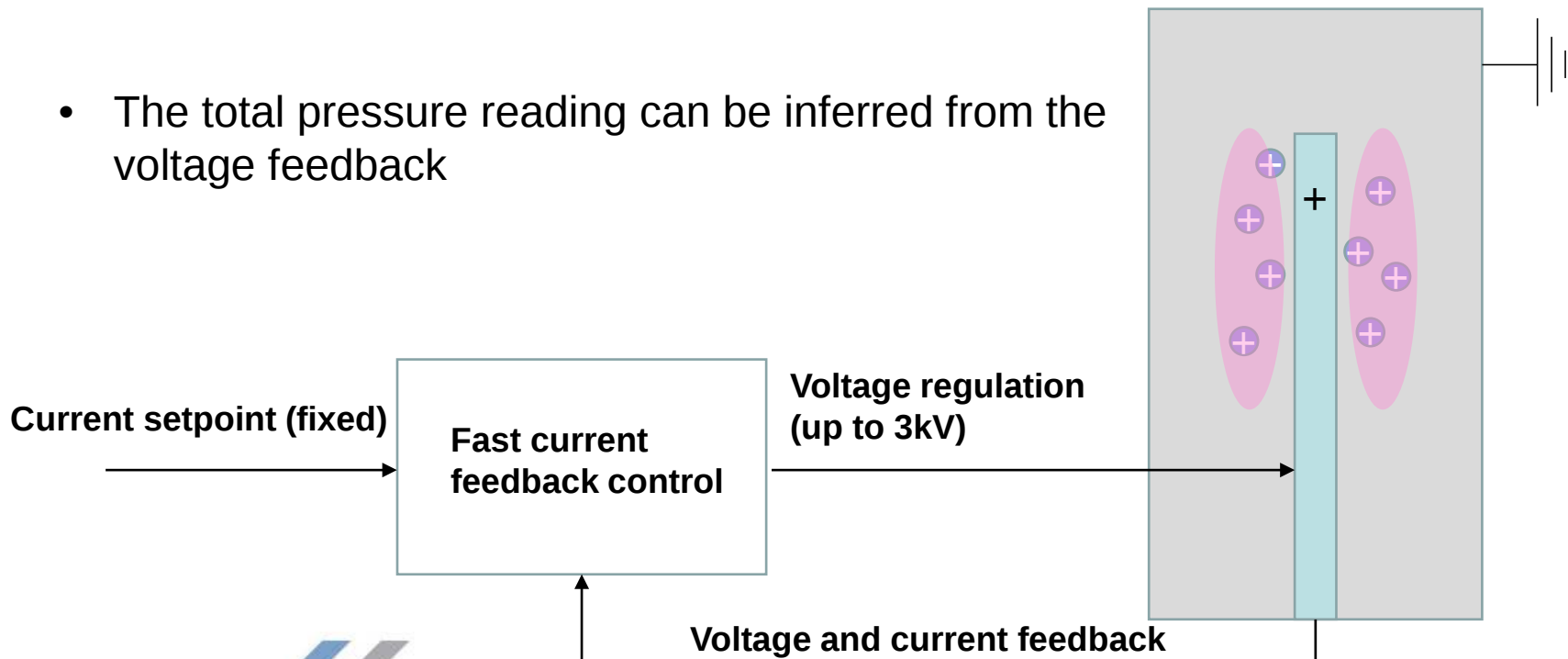


Miniature spectrometer



Plasma generation principle

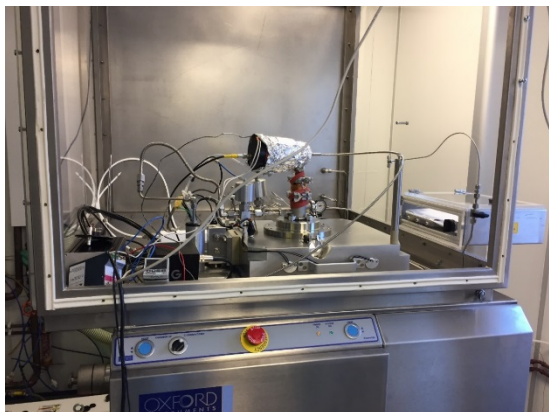
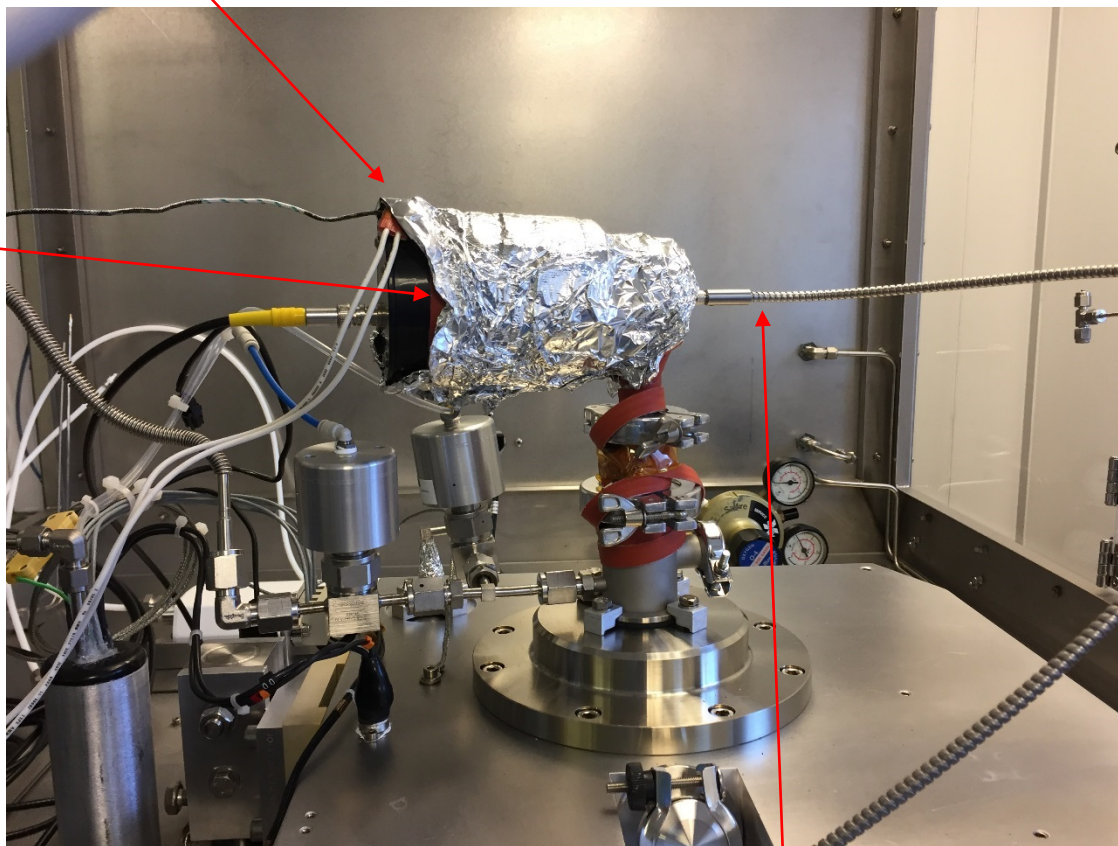
- Fast feedback control of the current allows for a stable plasma to be generated from **1E-6 mbar to 0.5 mbar**
- Overall species excitation is determine by the current setpoint.
- The total pressure reading can be inferred from the voltage feedback



Experimental setup

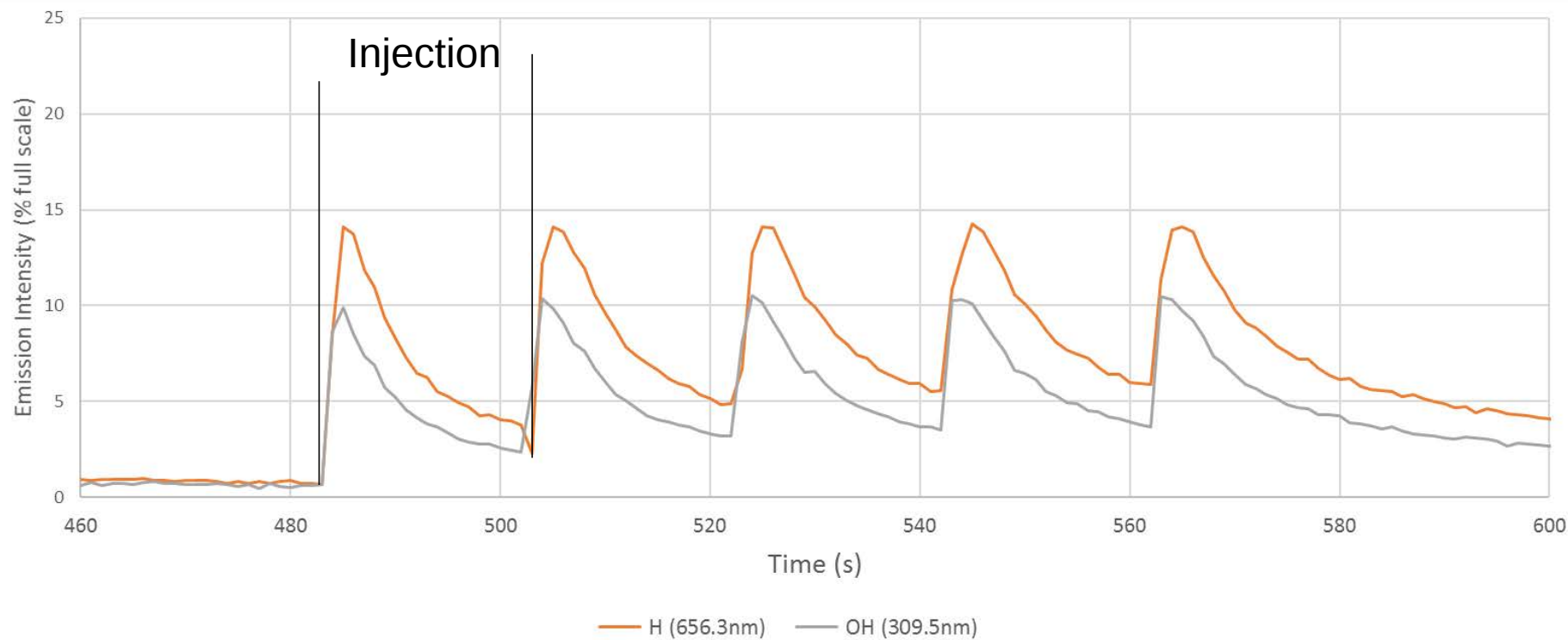
Thermocouple (100C setpoint)

Tape heater (240 V)



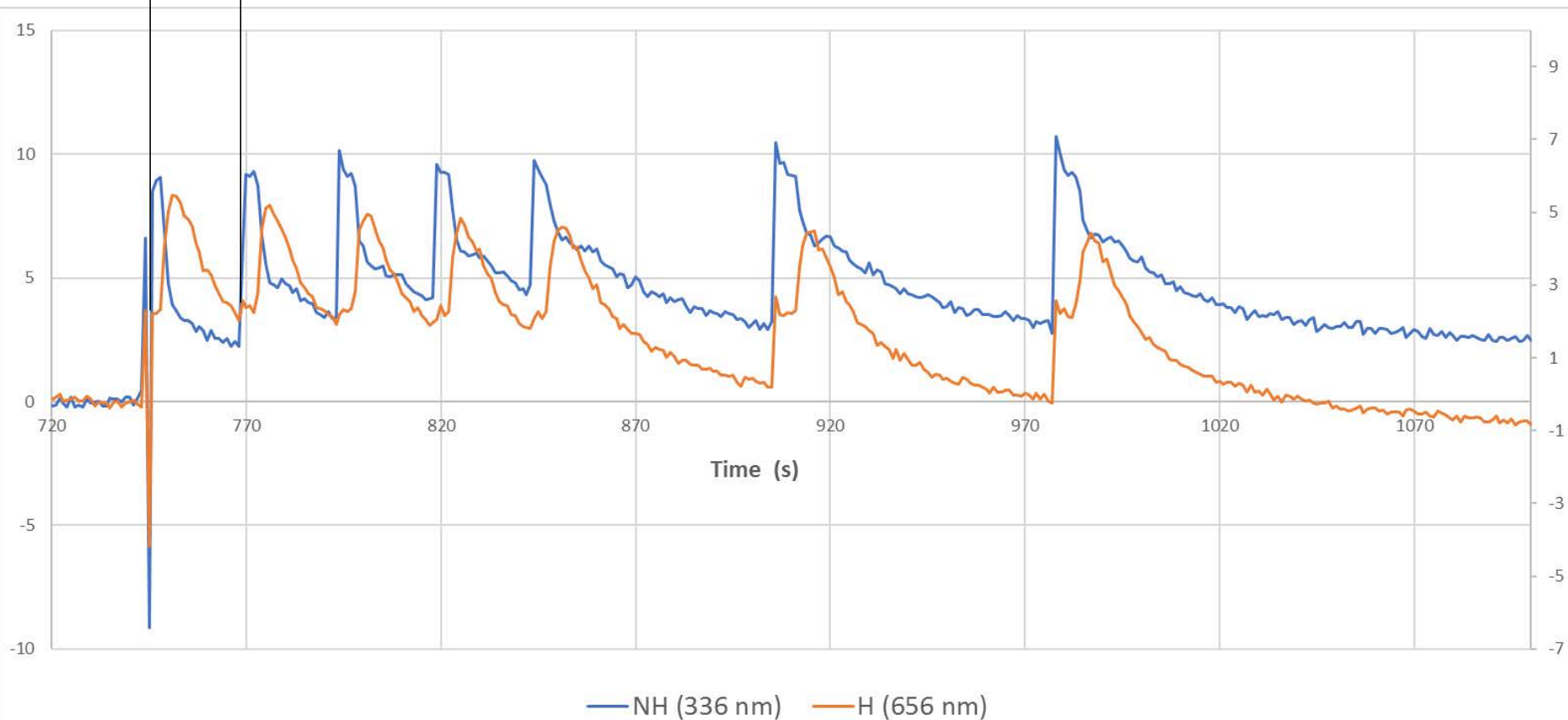
Fibre optic connection to spectrometer 6

Water vapour

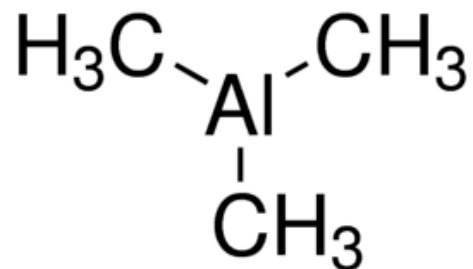


NH₃

Injection

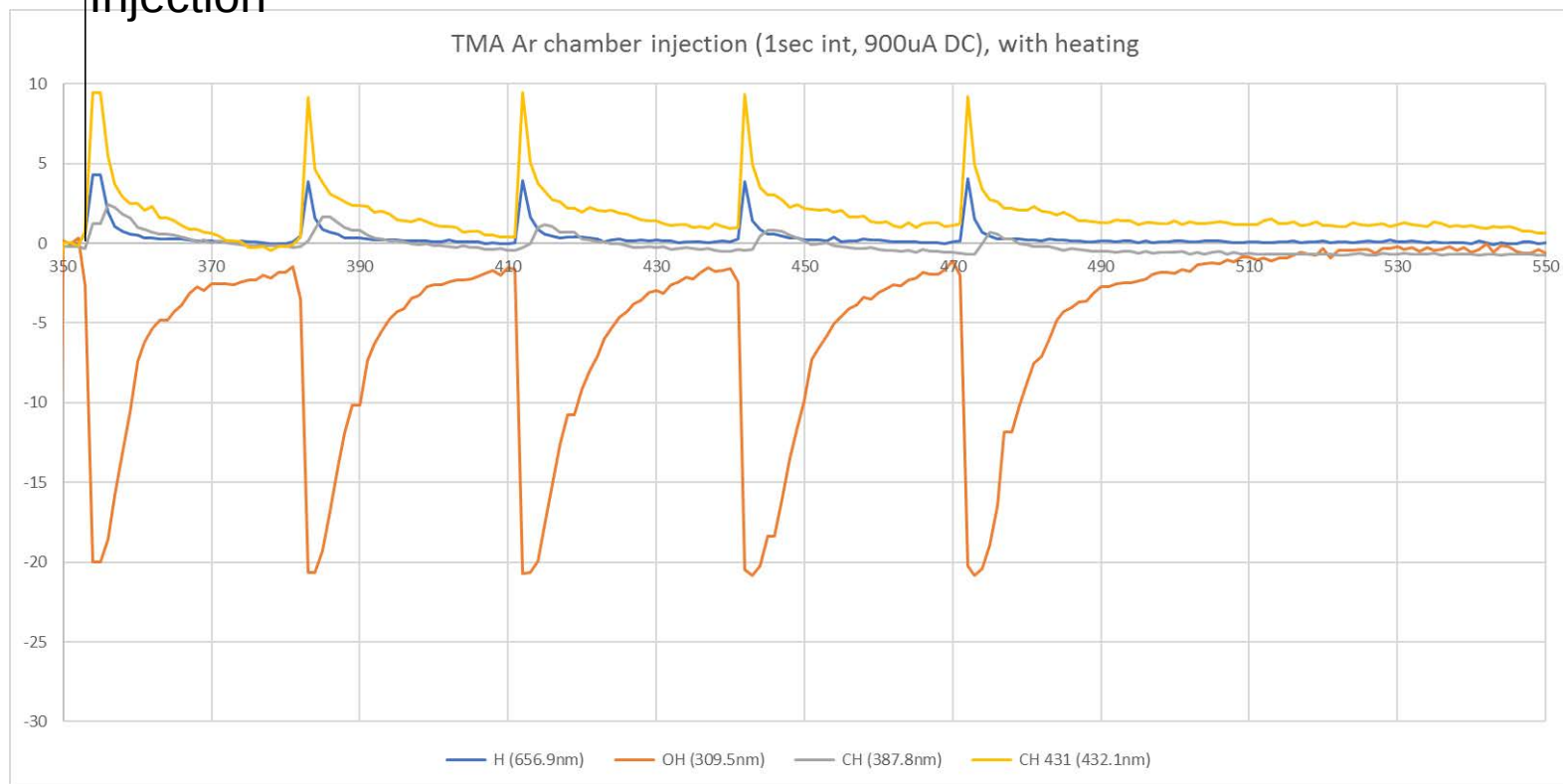


ALD Monitoring – Precursor detection



TMA

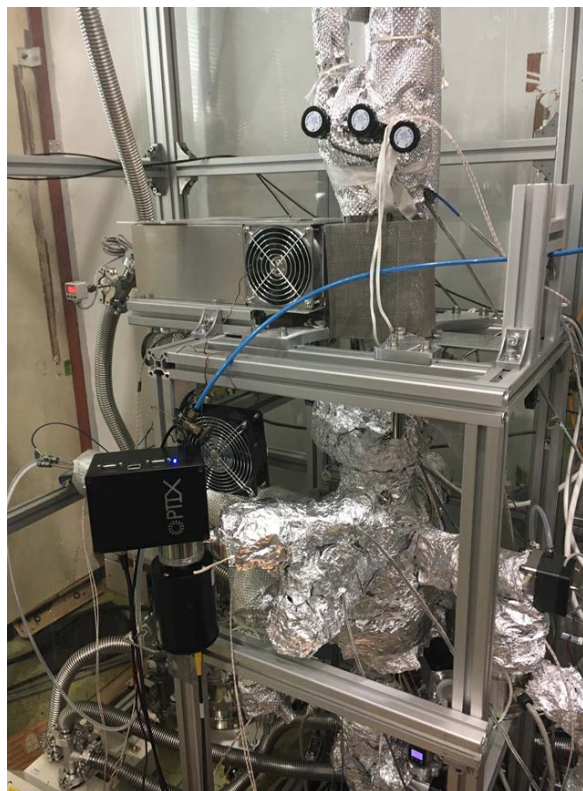
Injection



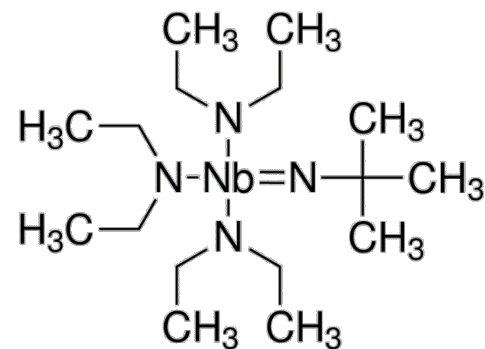
Deposition of NbN via PEALD

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

Image and data courtesy of S. Kato



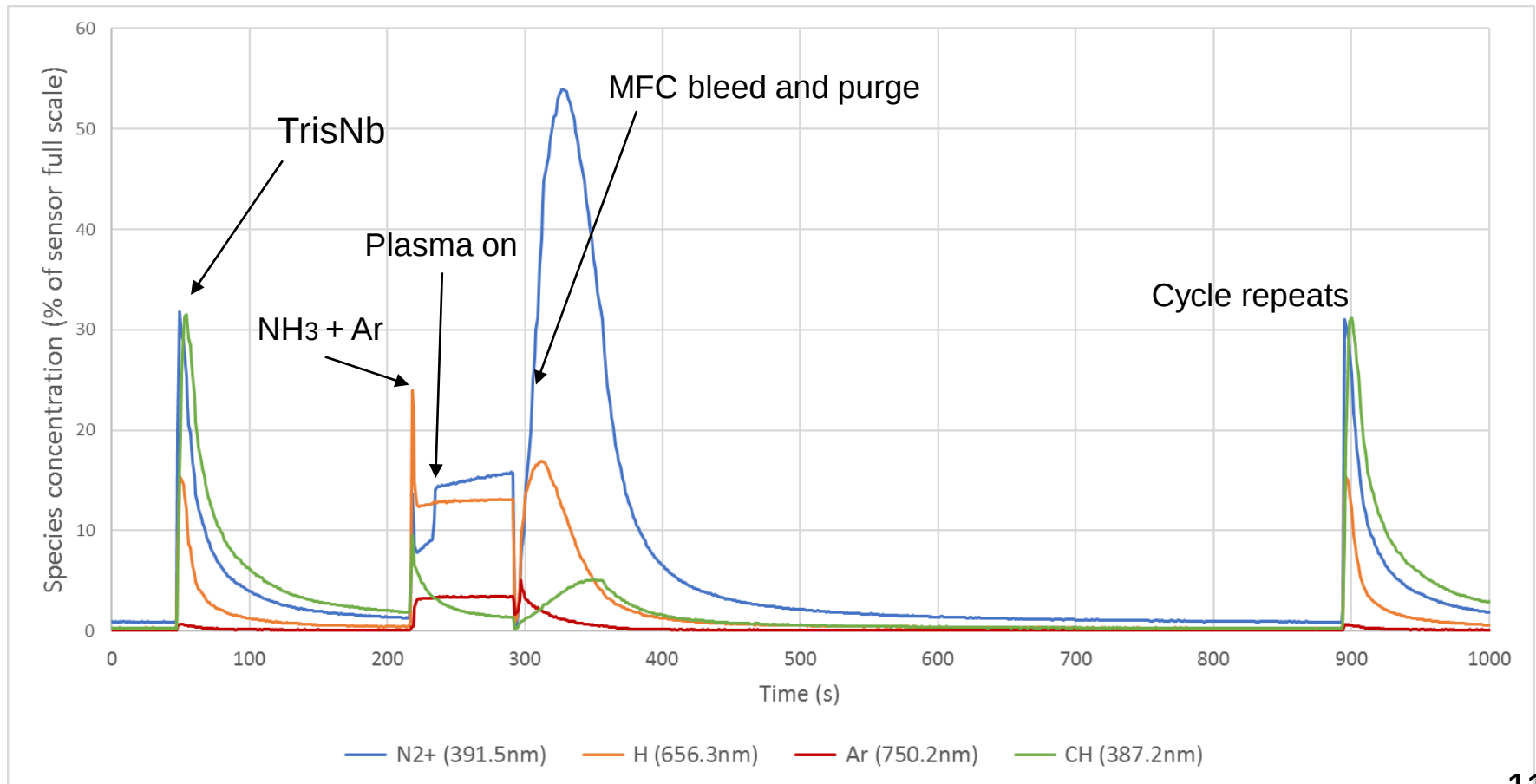
- Detection of TrisNb via CH, N and H



- Detection of NH₃ via N and H

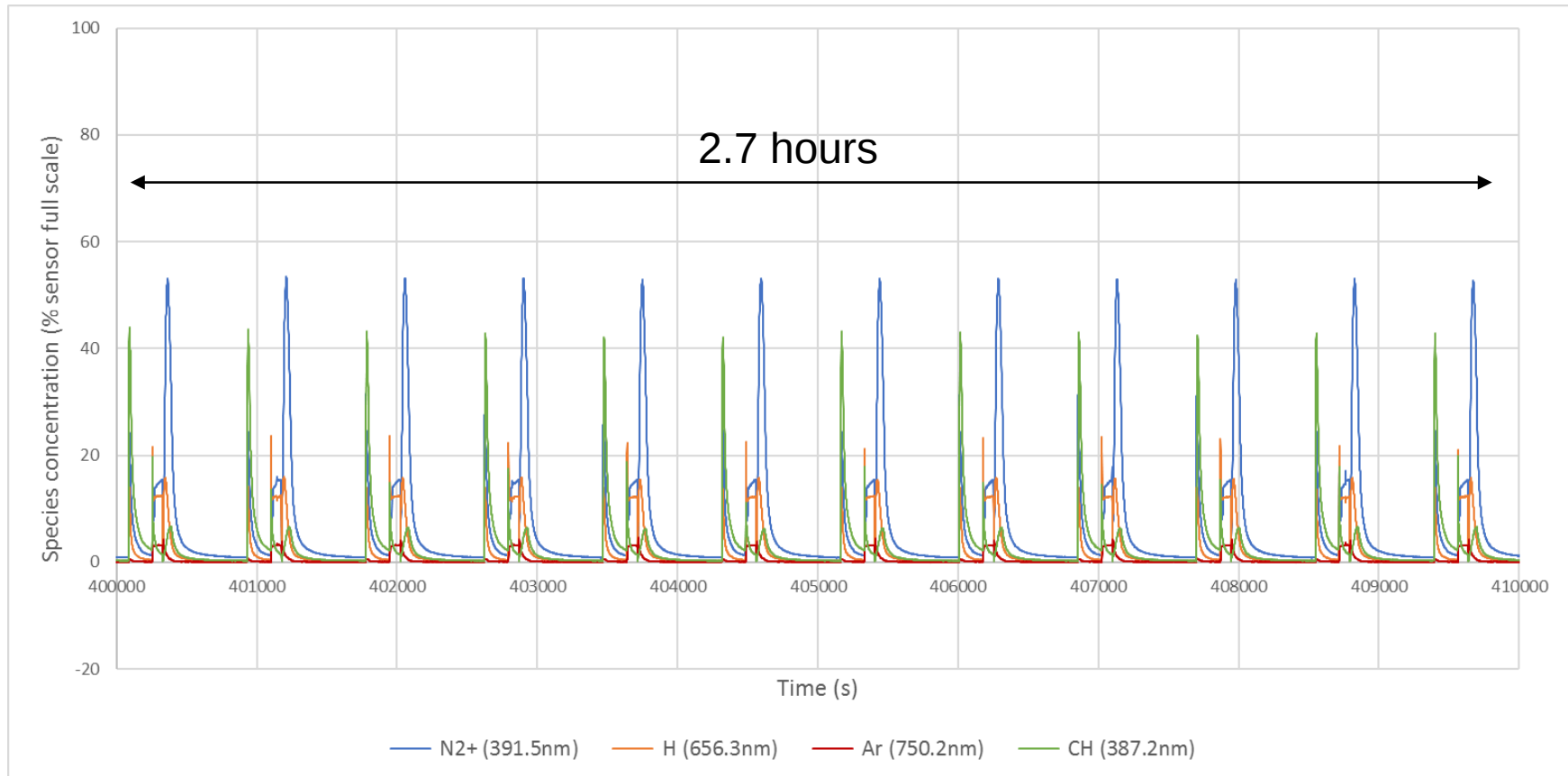
ALD Monitoring – Full process

NbN deposition step



ALD Monitoring – Full process

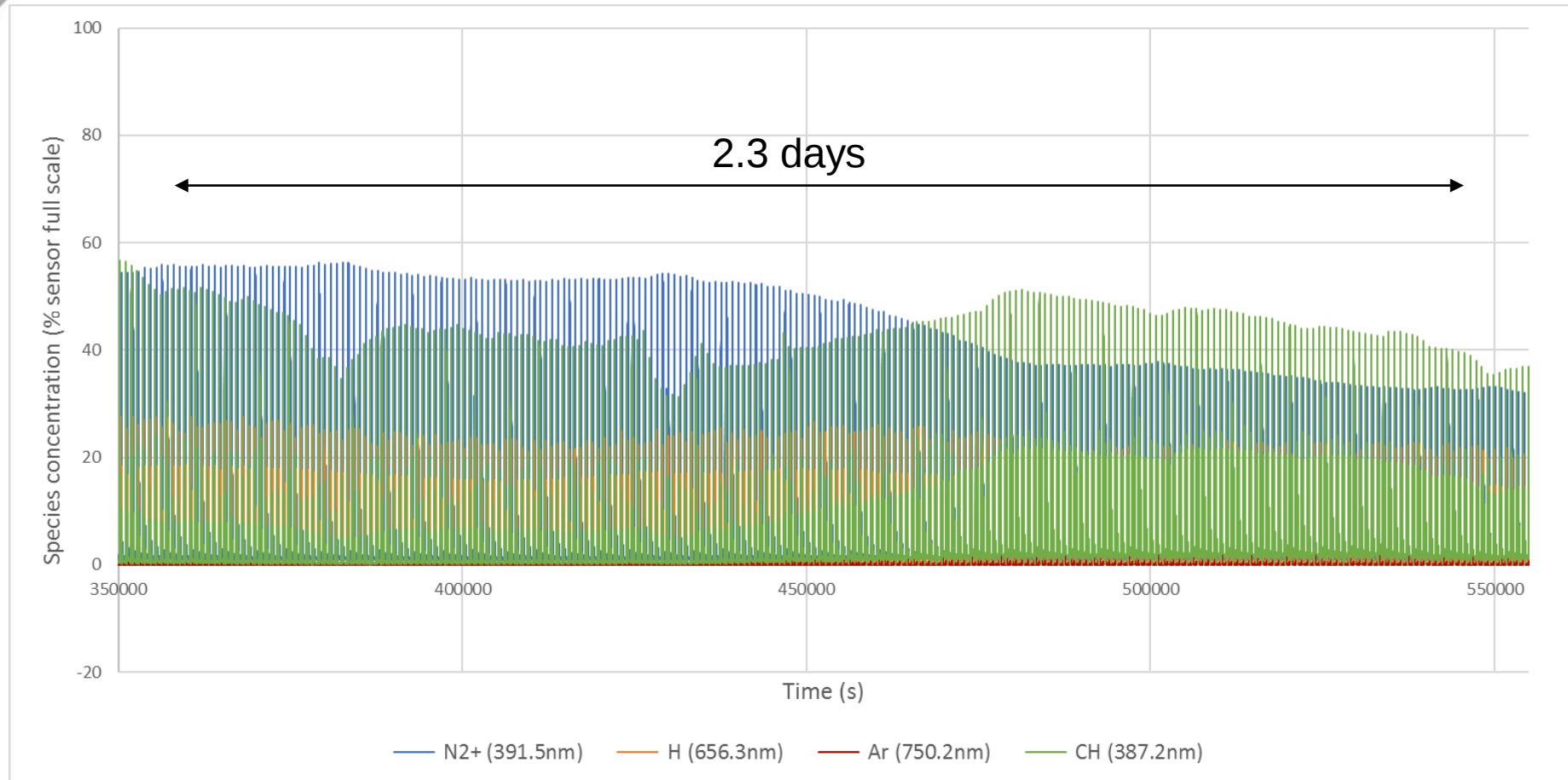
Deposition of NbN via PEALD



ALD Monitoring – Full process

Deposition of NbN via PEALD

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



Characterising an AlOx magnetron sputter deposition on roll-to-roll web

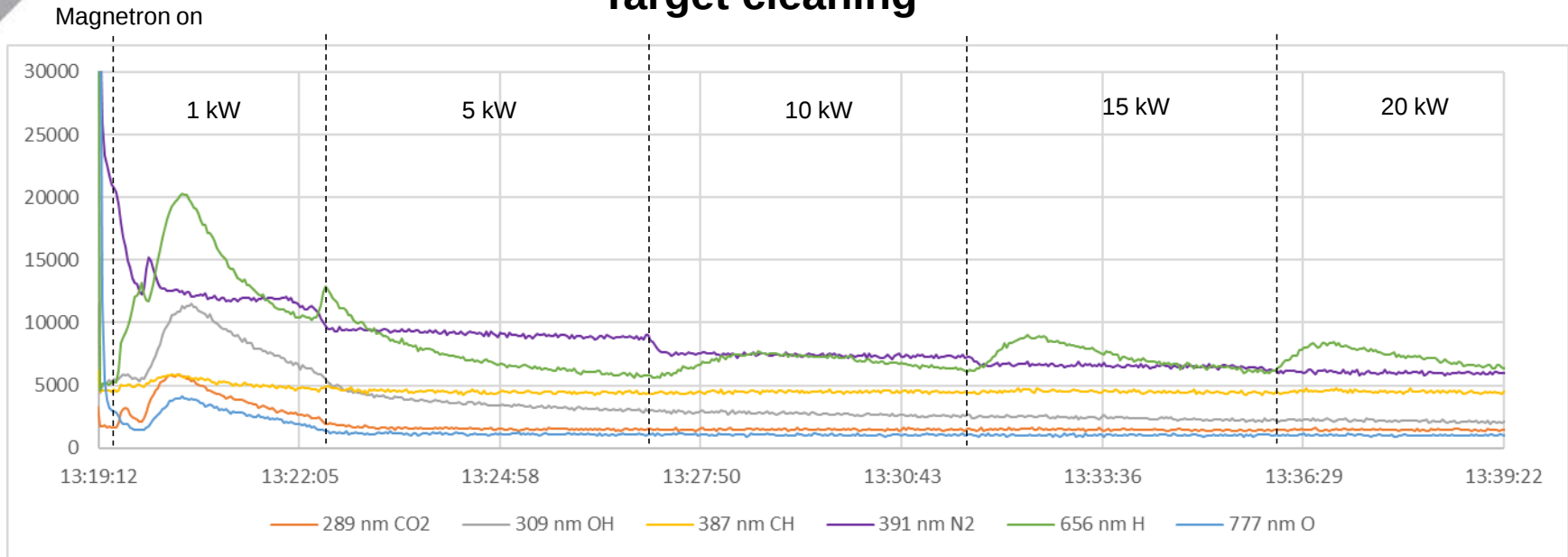
- Roll-to-roll deposition of reactively sputtered AlOx onto 125μm PET
- Optix sensor teed with a differentially pumped RGA



Courtesy of Emerson and Renwick

Characterising an AlOx magnetron sputter deposition on roll-to-roll web

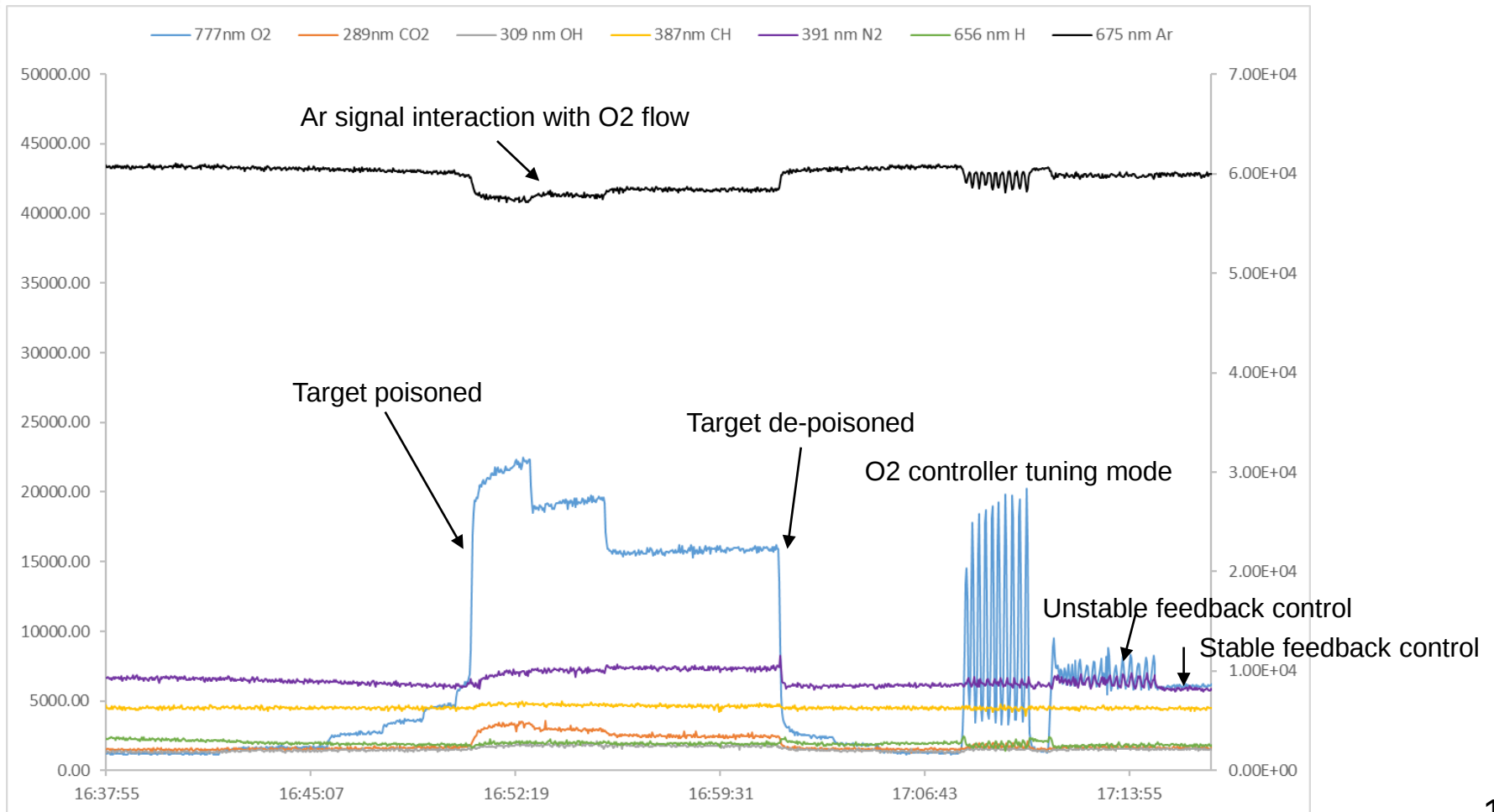
Target cleaning



- 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

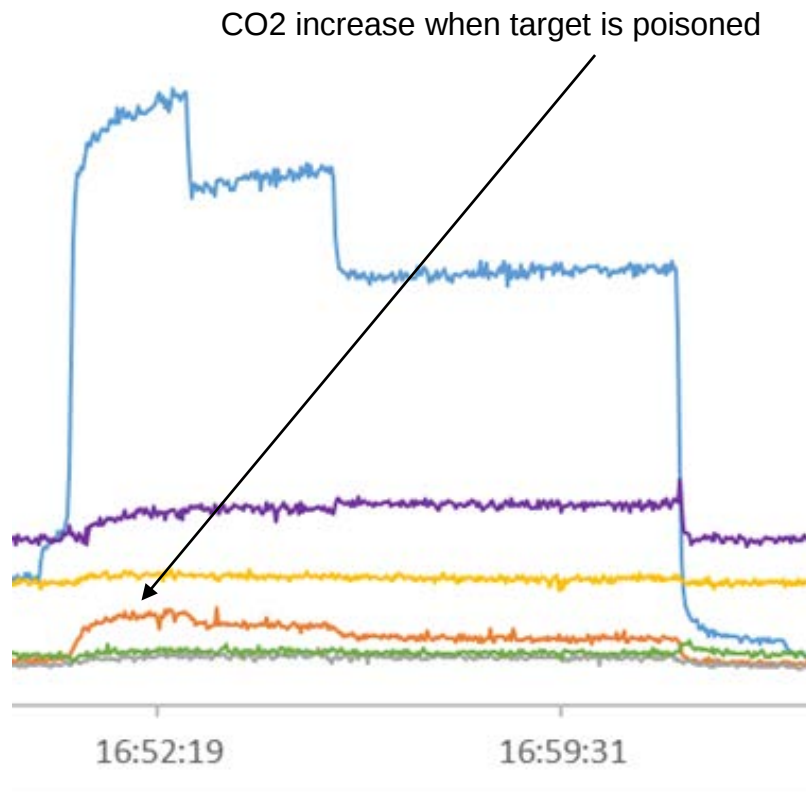
Characterising an AlOx magnetron sputter deposition on roll-to-roll web

Reactive sputter characterisation



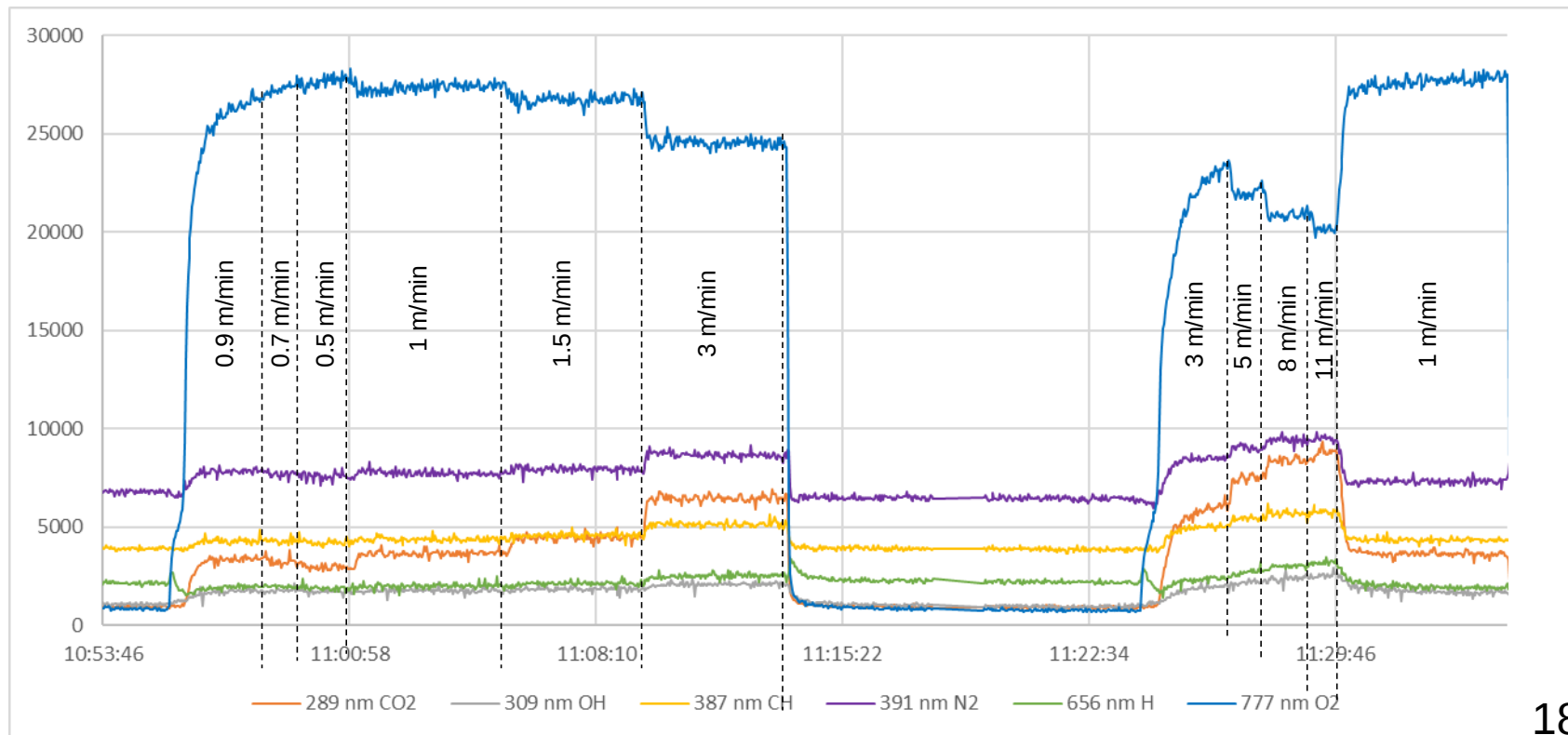
Characterising an AlOx magnetron sputter deposition on roll-to-roll web

- There is a clear increase in CO₂ observed when the target becomes poisoned



Characterising an AlOx magnetron sputter deposition on roll-to-roll web

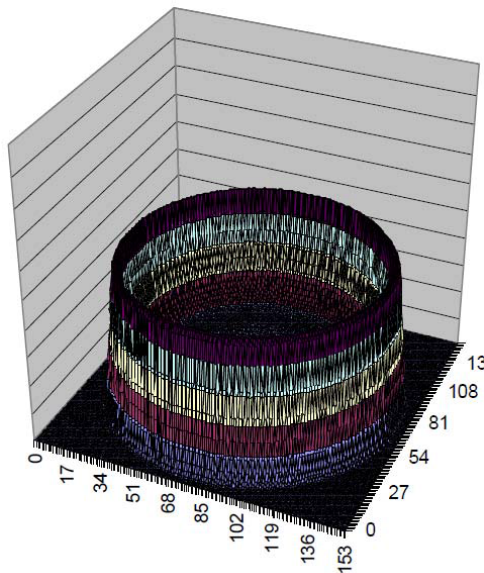
- Web speed varied to determine the source of the CO₂
 - No influence = cathode
 - Influence = web
- Strong influence of web speed observed
- Inverse effect on O₂ observed



Characterising an AlOx magnetron sputter deposition on roll-to-roll web

Problem summary

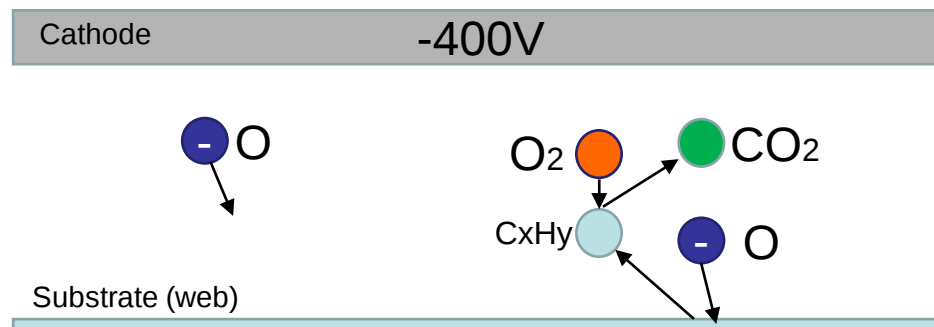
- Main species observed developing are CO₂ and N₂
- Only occurs when target is poisoned
- Web speed influences the observed concentration



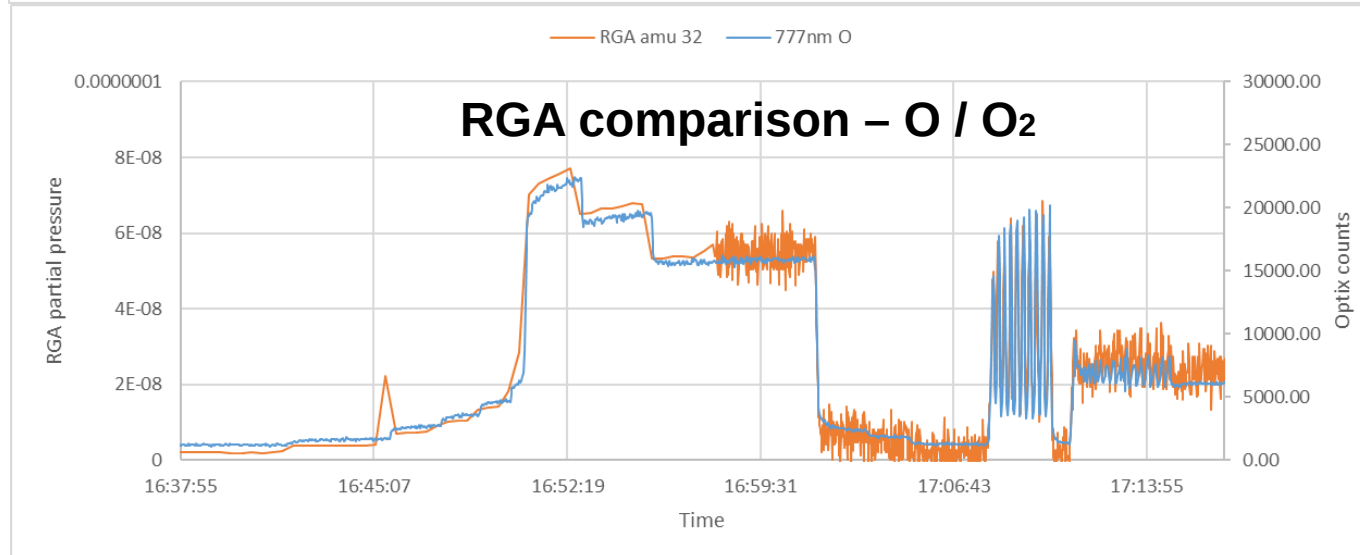
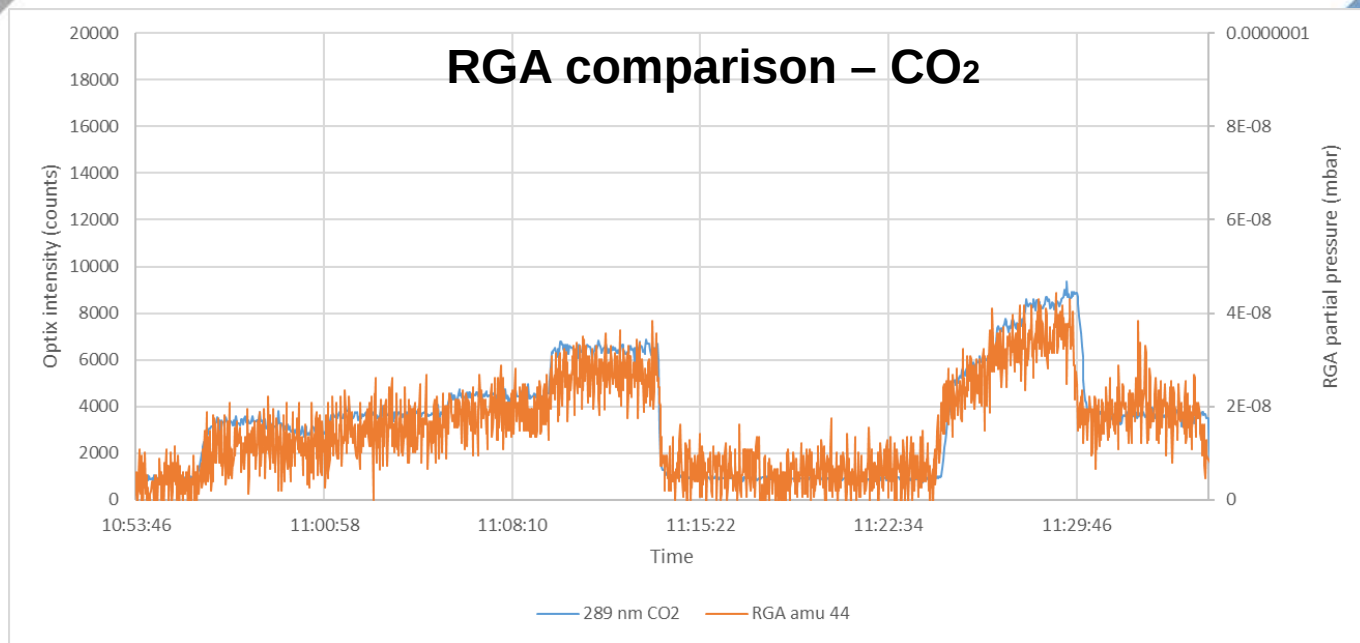
Simulation of (-) O ions emanating from poisoned cathode

Hypotheses

- Negative oxygen ion bombardment of the substrate occurs when the target (cathode) becomes poisoned
- In this case, the bombardment can be liberating species from the film surface. This is a mix of hydrocarbons and nitrogen
- The hydrocarbons are reacting with the activated oxygen process gas to form CO₂

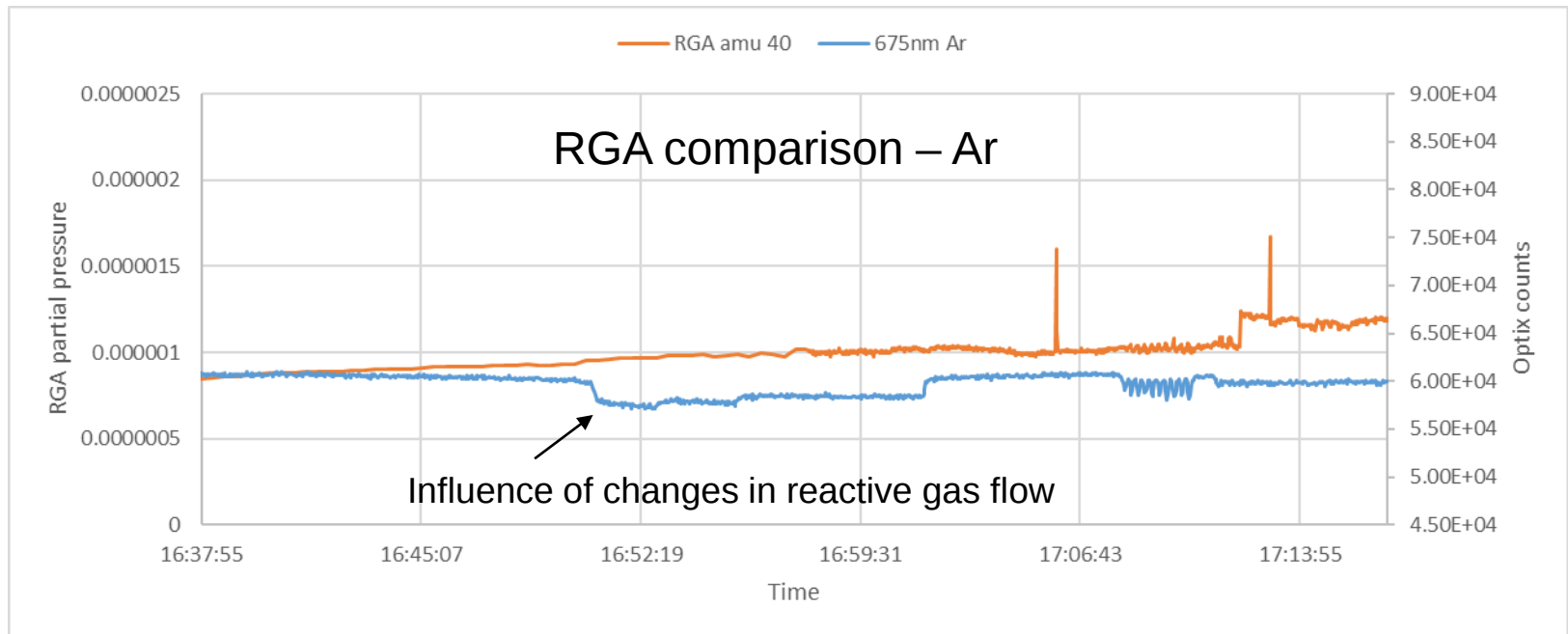


Characterising an AlOx magnetron sputter deposition on roll-to-roll web



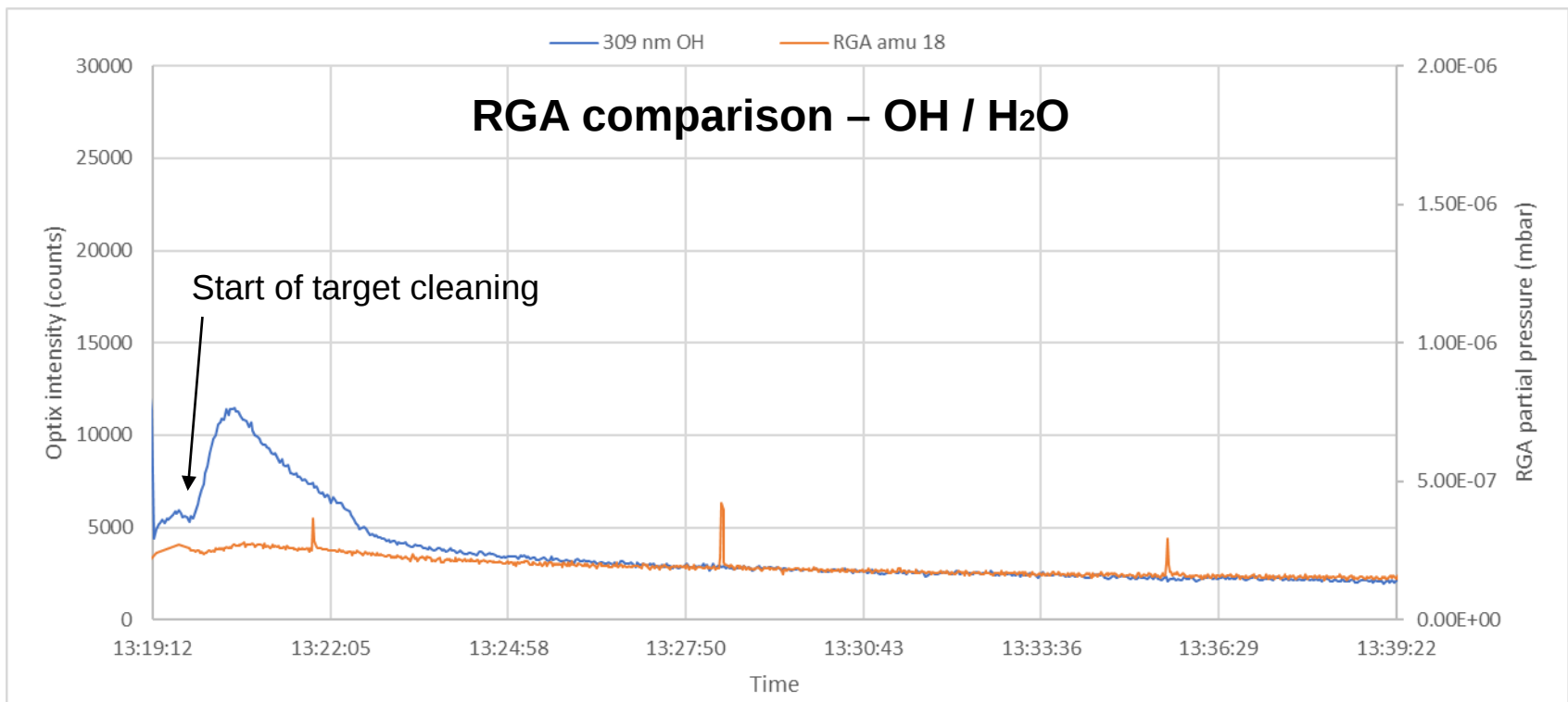
Characterising an AlOx magnetron sputter deposition on roll-to-roll web

- 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

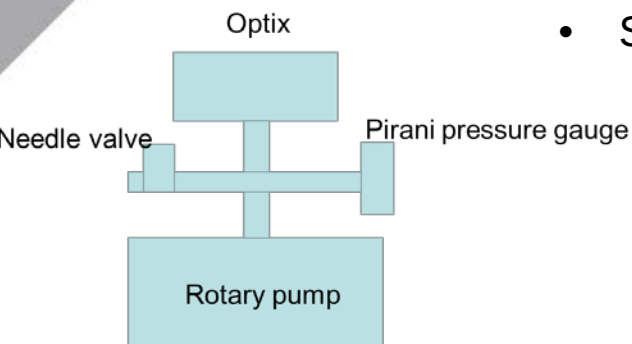


Characterising an AlOx magnetron sputter deposition on roll-to-roll web

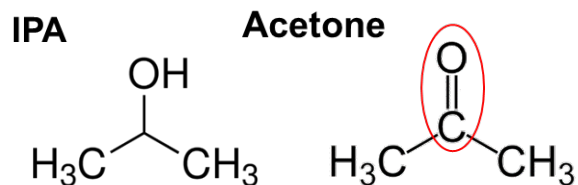
- Large difference in the water vapour reading
- Higher sensitivity for Optix sensor due to absence of differential sampling system



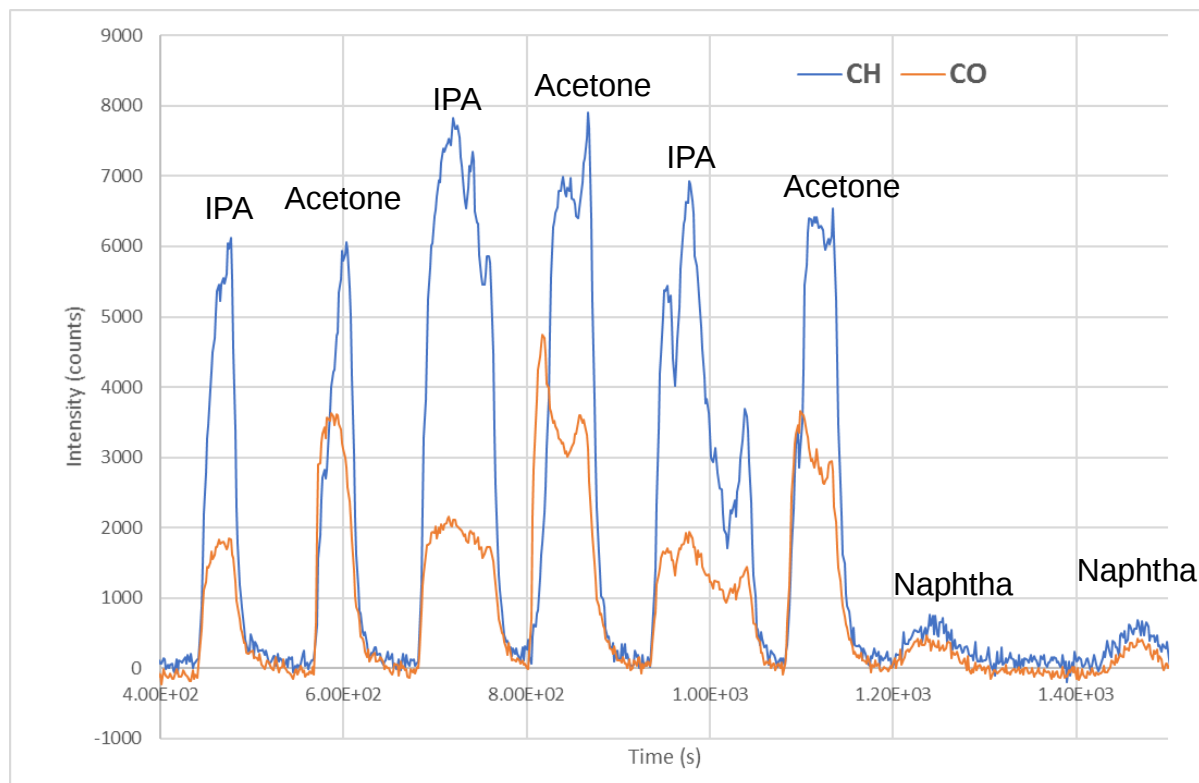
Detection at atmospheric pressures



- Simple setup allows use at atmospheric (and above) pressures



Acetone has a higher CO reading due to the presence of a CO bond



Conclusions

- Remote PEM (RPEM) sensing technique can be used to directly monitor many roll to roll vacuum processes
- Results are comparable to a diff. pumped RGA and are in some cases (H_2O) more sensitive
- Robustness has been demonstrated with contaminating processes, such as ALD