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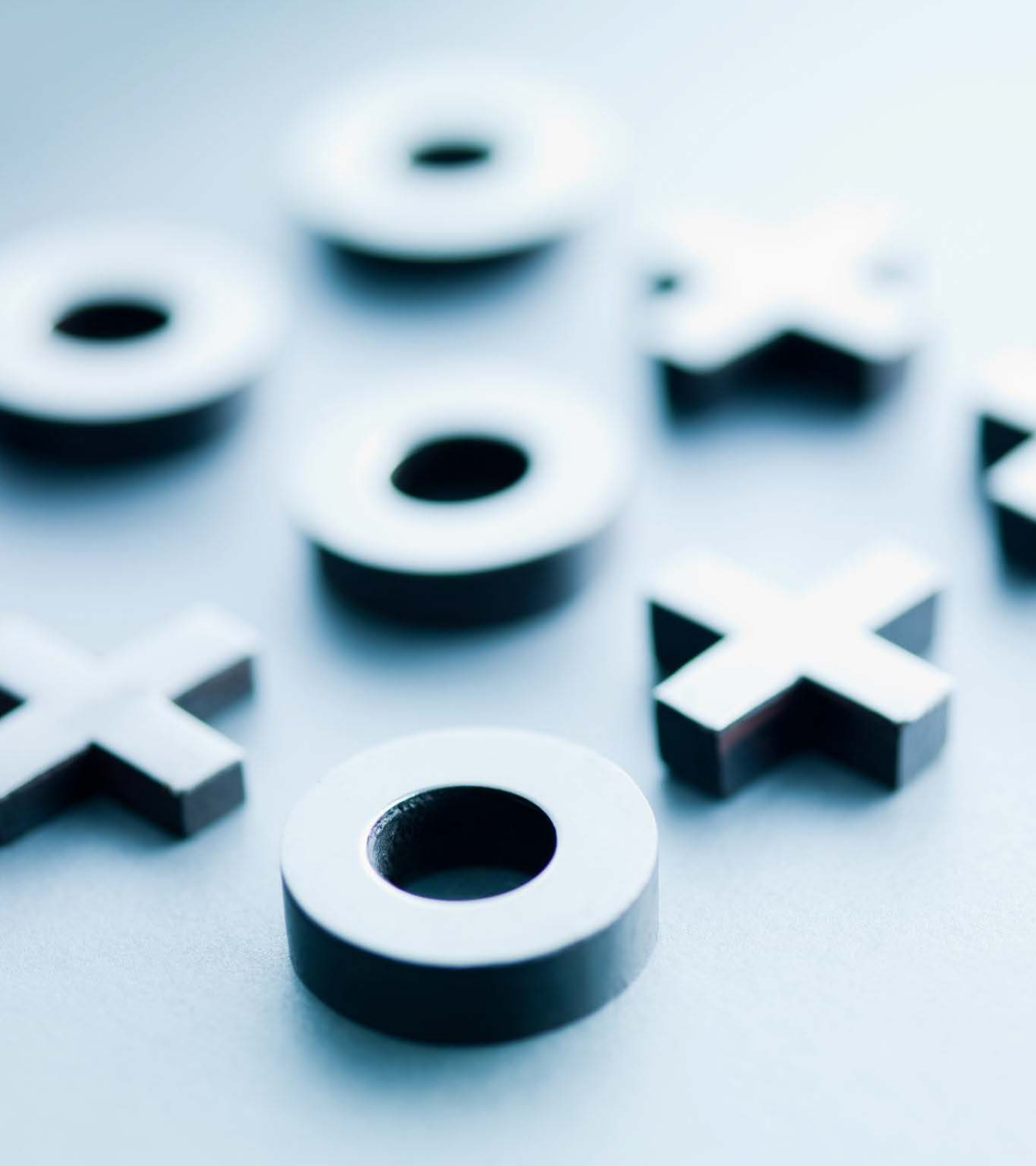
# NH<sub>3</sub> – High temperature experimentation

Vegar Øygarden, Belma Talic, SINTEF

NH<sub>3</sub> webinar, AEGIR, 14.02.2023



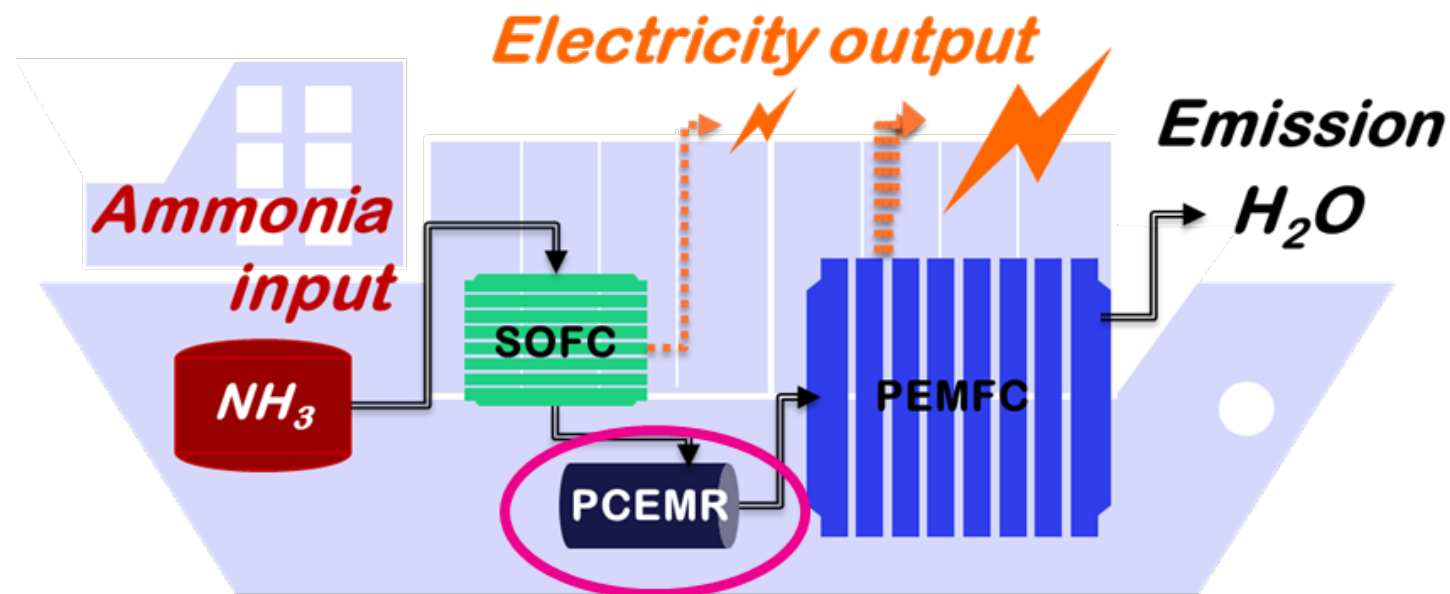
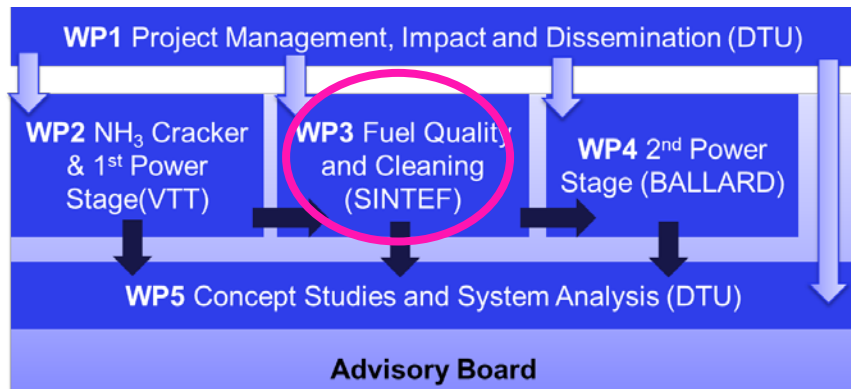
Teknologi for et bedre samfunn



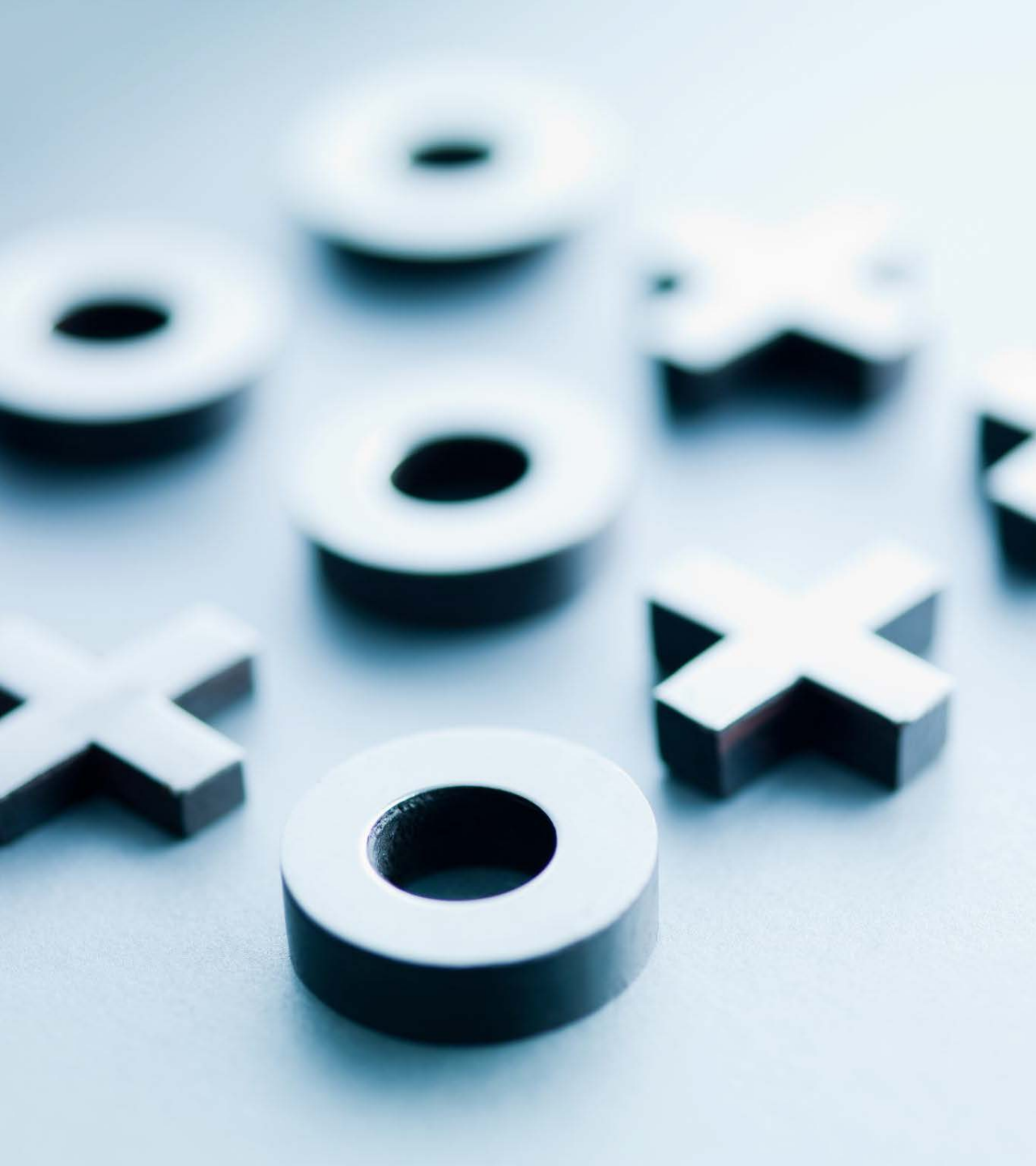
## Outline

- Short introduction
- Working with  $\text{NH}_3$  at high temperatures
  - Prior to experiment
  - During experiment
  - After experiment
- Purpose-built  $\text{NH}_3$ -lab

# AEGIR project



PCEMR – Proton Ceramic Electrochemical Membrane Reactor  
 Extraction of high purity H<sub>2</sub> from NH<sub>3</sub>/H<sub>2</sub>/N<sub>2</sub> mix



## Outline

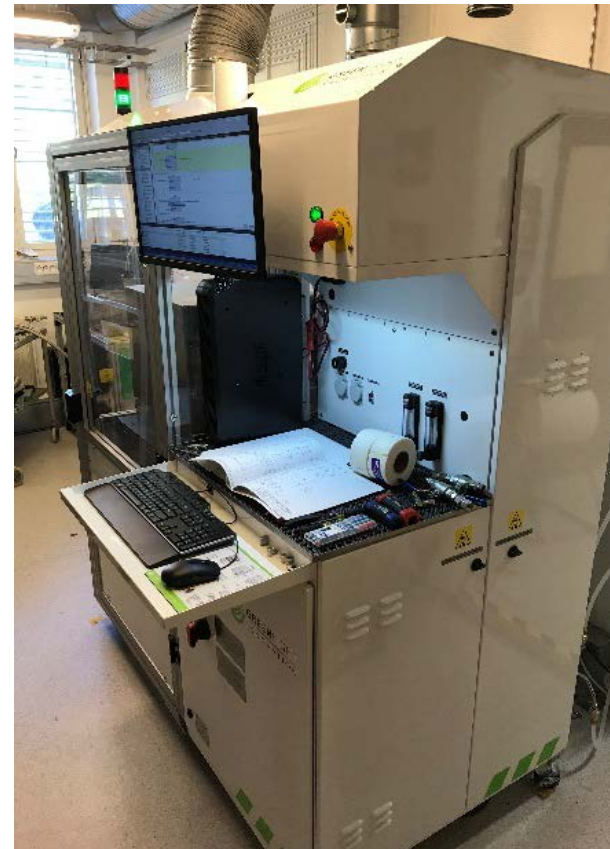
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# Prior to experimental start-up start

- All valves, gaskets and MFCs in existing infrastructure was replaced to qualify for use of concentrated dry/wet  $\text{NH}_3$ .
- Safety assessment of the  $\text{NH}_3$  activities, following standard procedures for safe job analysis at SINTEF.
- FTIR used for gas analysis.
- Scrubbing system for exhaust.
- Bubbling through saturated  $\text{Cu}(\text{NO}_3)_2$ -solution
  - $\text{Cu}(\text{NO}_3)_2 + \text{NH}_3 = \text{Cu}(\text{OH})_{2(s)} + \text{Cu}(\text{NH}_3)_4^{2+}$
  - $\text{Cu}(\text{OH})_{2(s)}$  can cause clogging of gas lines.
- Bubbling through lactic acid
  - Liquid ammonium complex
  - Keep track of pH





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# NH<sub>3</sub>-cracking – Choice of gas-inlet material

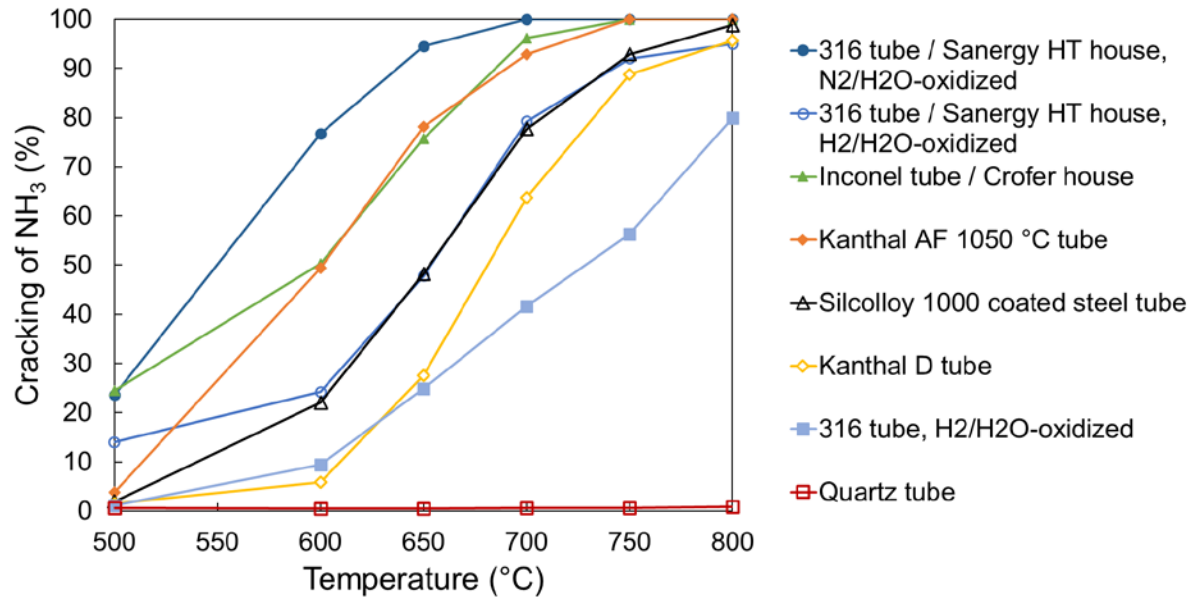


Figure 2: Ammonia cracking as a function of temperature for different materials utilizing a flow rate of 0.12 L/min 10% NH<sub>3</sub>/Ar.

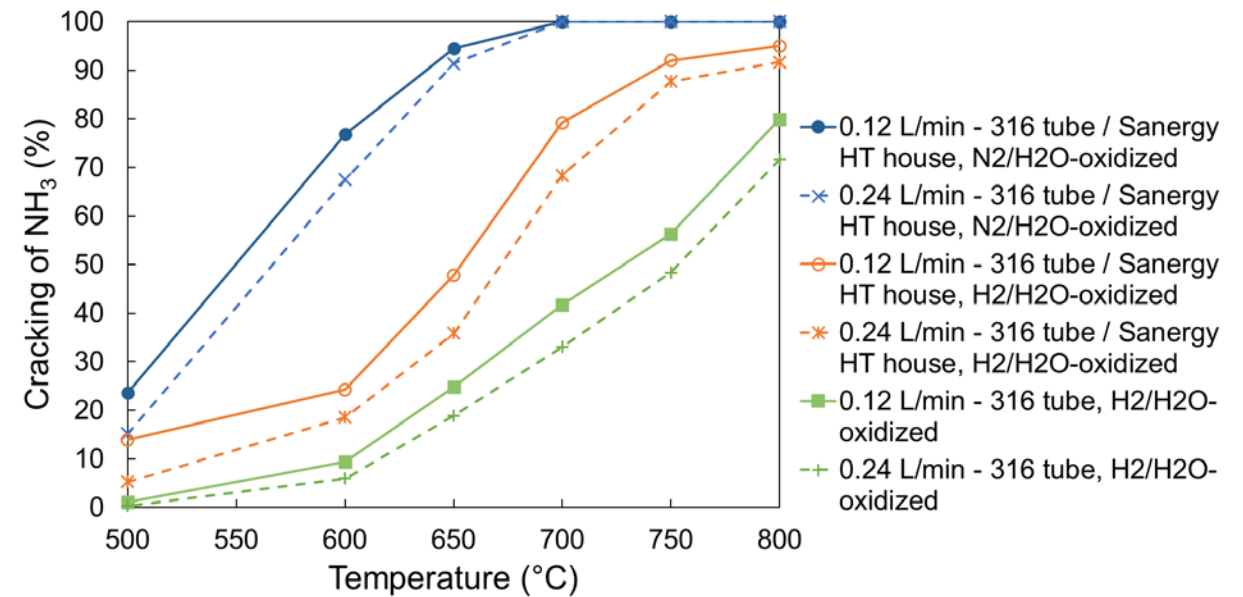
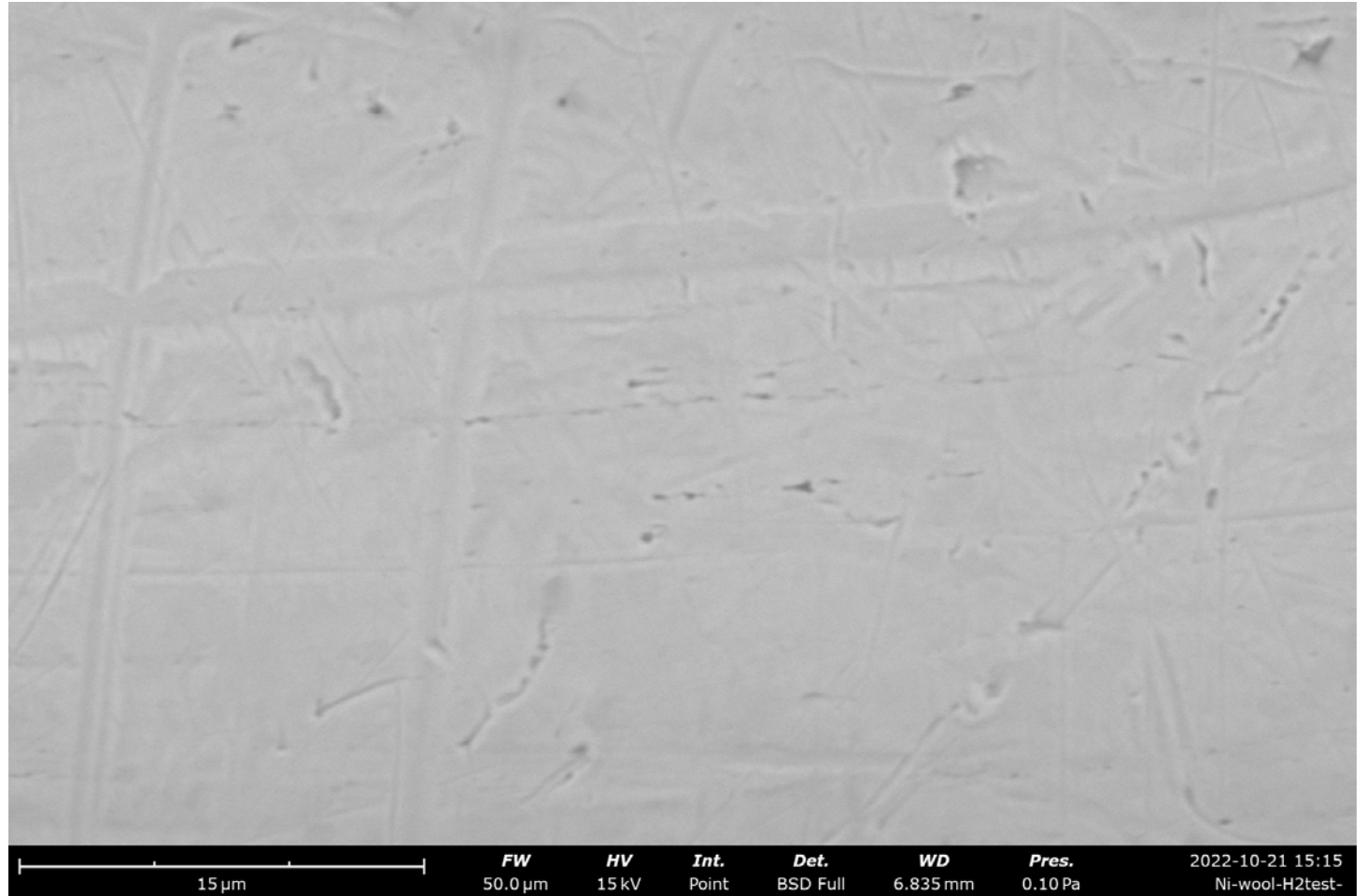
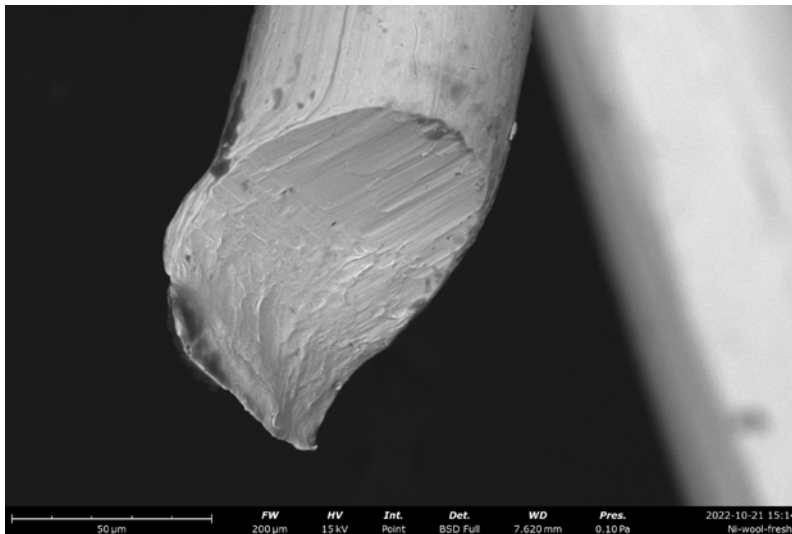
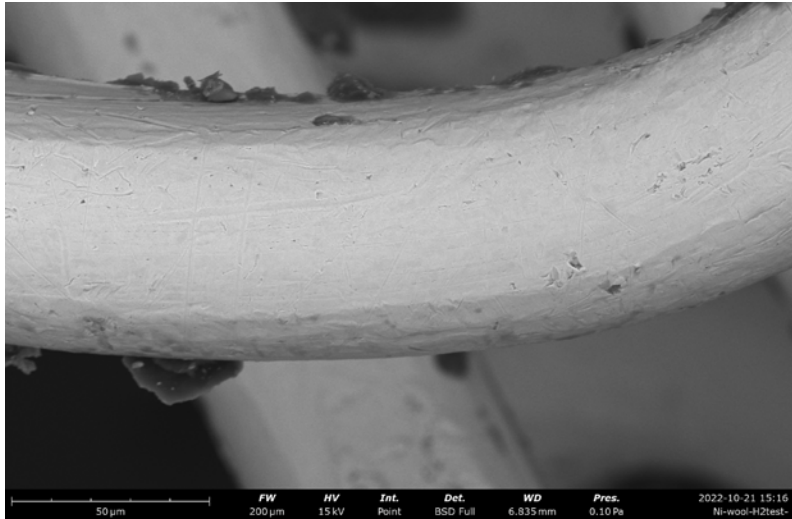


Figure 3: Ammonia cracking as a function of temperature for different materials utilizing a flow rate of 0.12 L/min (solid lines) or 0.24 L/min (dashed lines) of 10% NH<sub>3</sub>/Ar.



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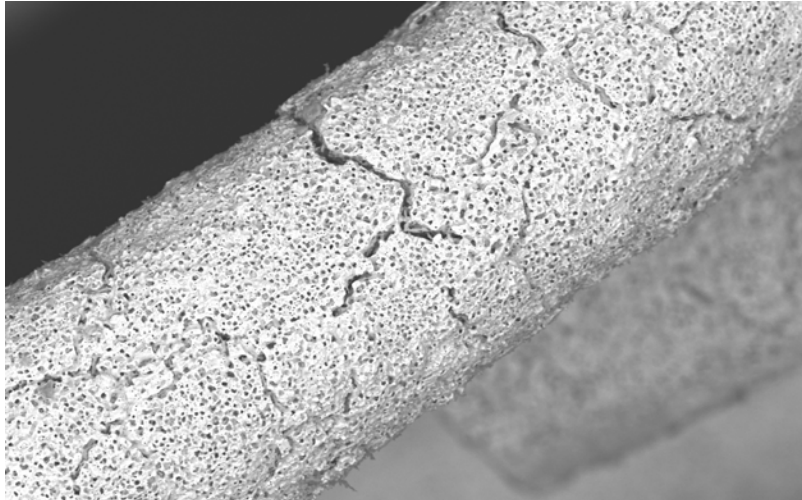
# Ni-wool from H<sub>2</sub> test



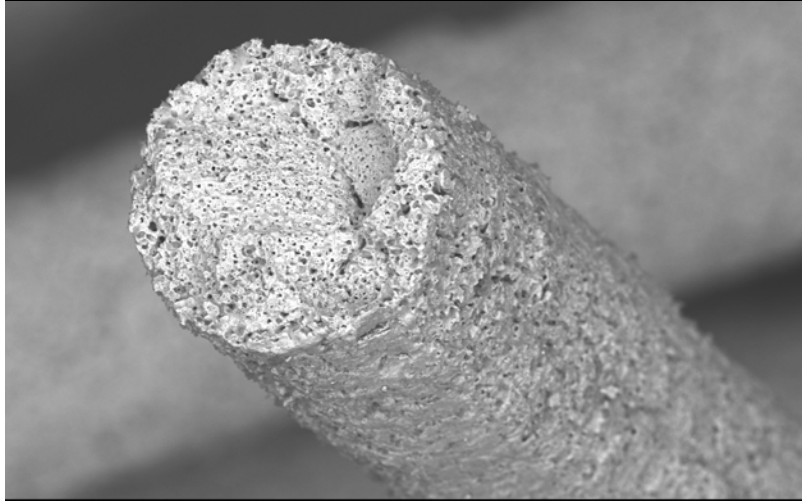


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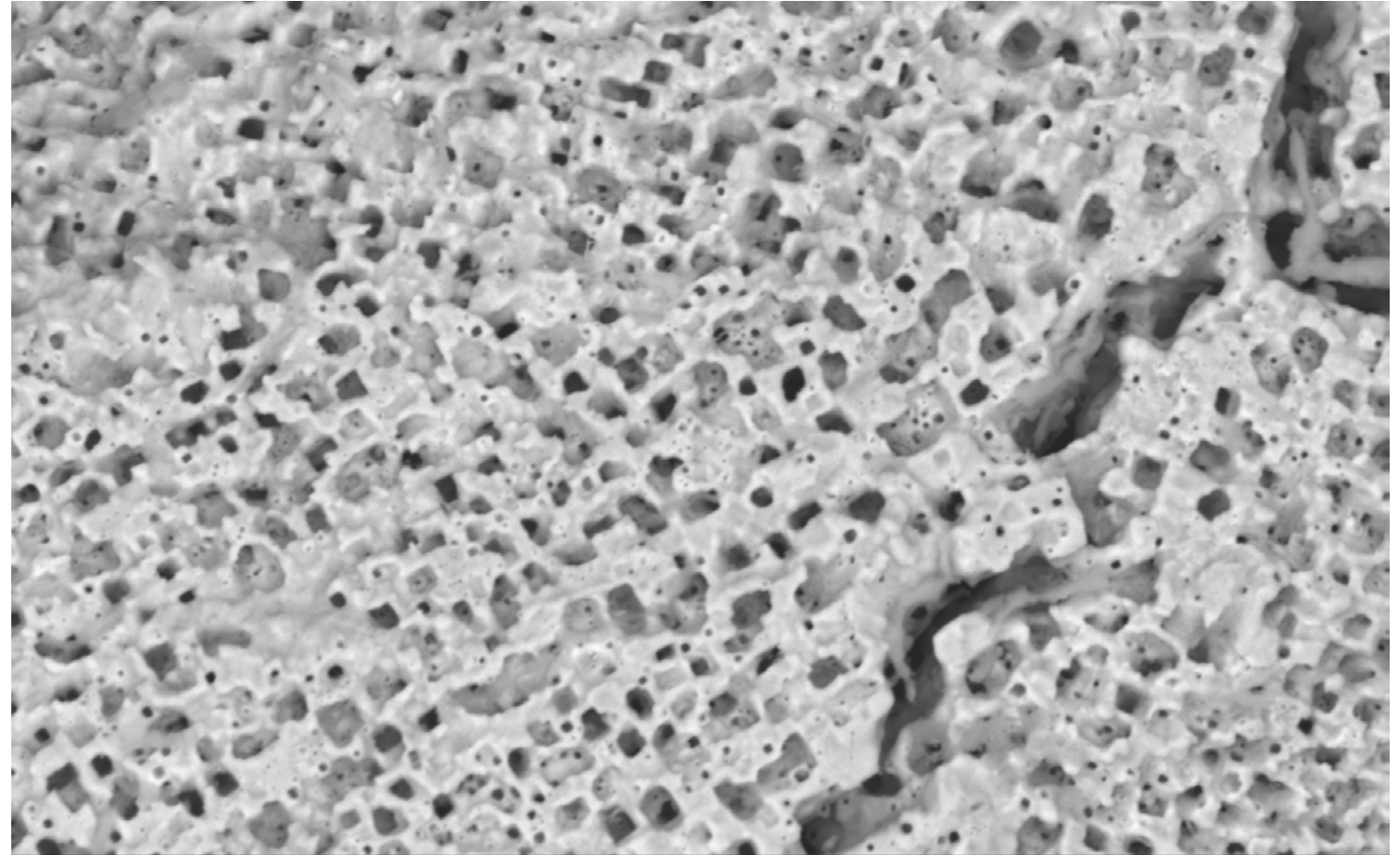
# Ni-wool from test with $\text{NH}_3$



50  $\mu\text{m}$  FW 200  $\mu\text{m}$  HV 15 kV Int. Point Det. BSD Full WD 9.385 mm Pres. 0.10 Pa 2022-10-12 15:51 Ni-wool-PCEMR-5-



50  $\mu\text{m}$  FW 200  $\mu\text{m}$  HV 15 kV Int. Point Det. BSD Full WD 9.581 mm Pres. 0.10 Pa 2022-10-12 15:50 Ni-wool-PCEMR-5-



15  $\mu\text{m}$  FW 50.0  $\mu\text{m}$  HV 15 kV Int. Point Det. BSD Full WD 9.385 mm Pres. 0.10 Pa 2022-10-12 15:52 Ni-wool-PCEMR-5-

→ 5-15 % increase in wire diameter

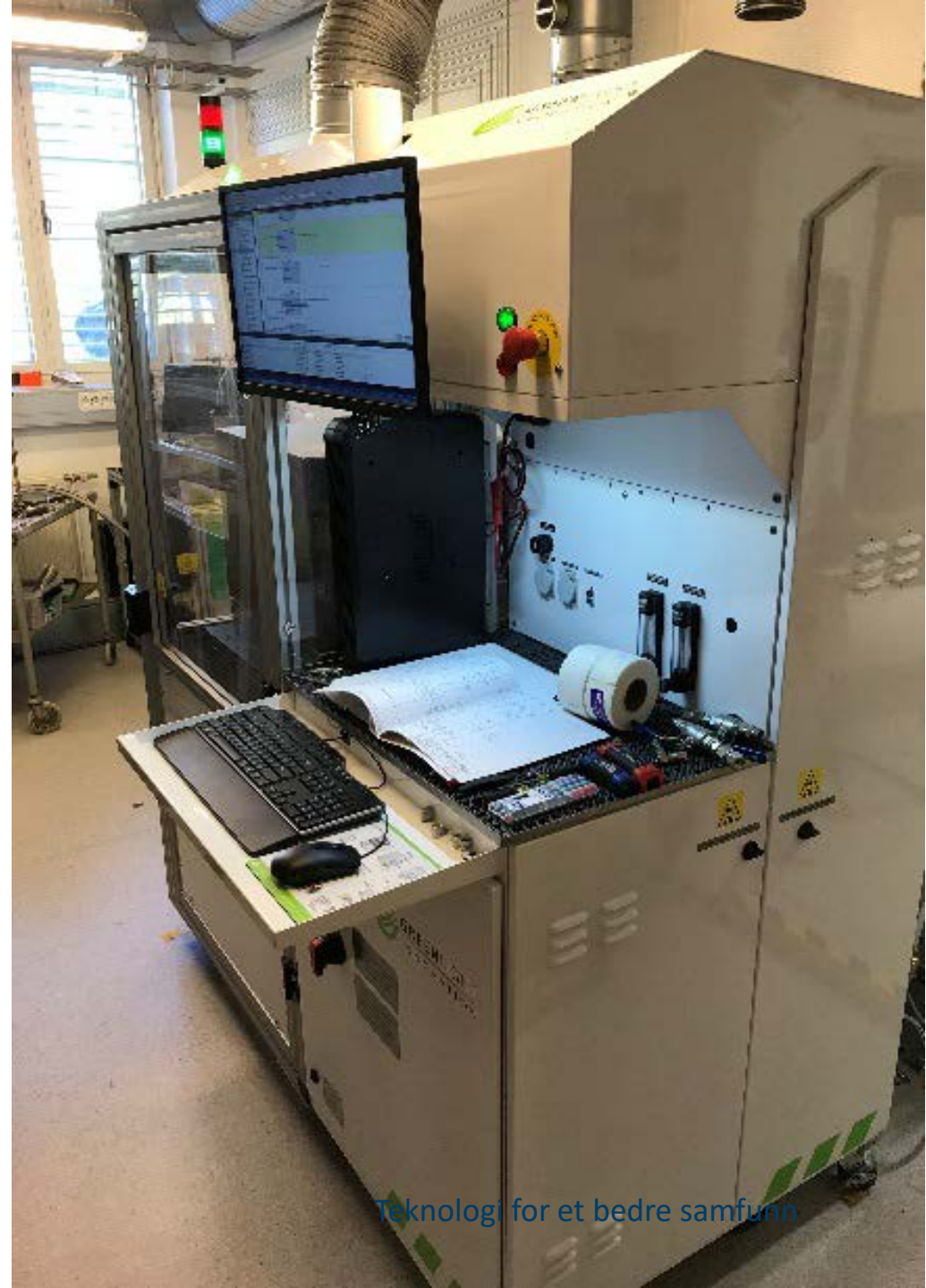


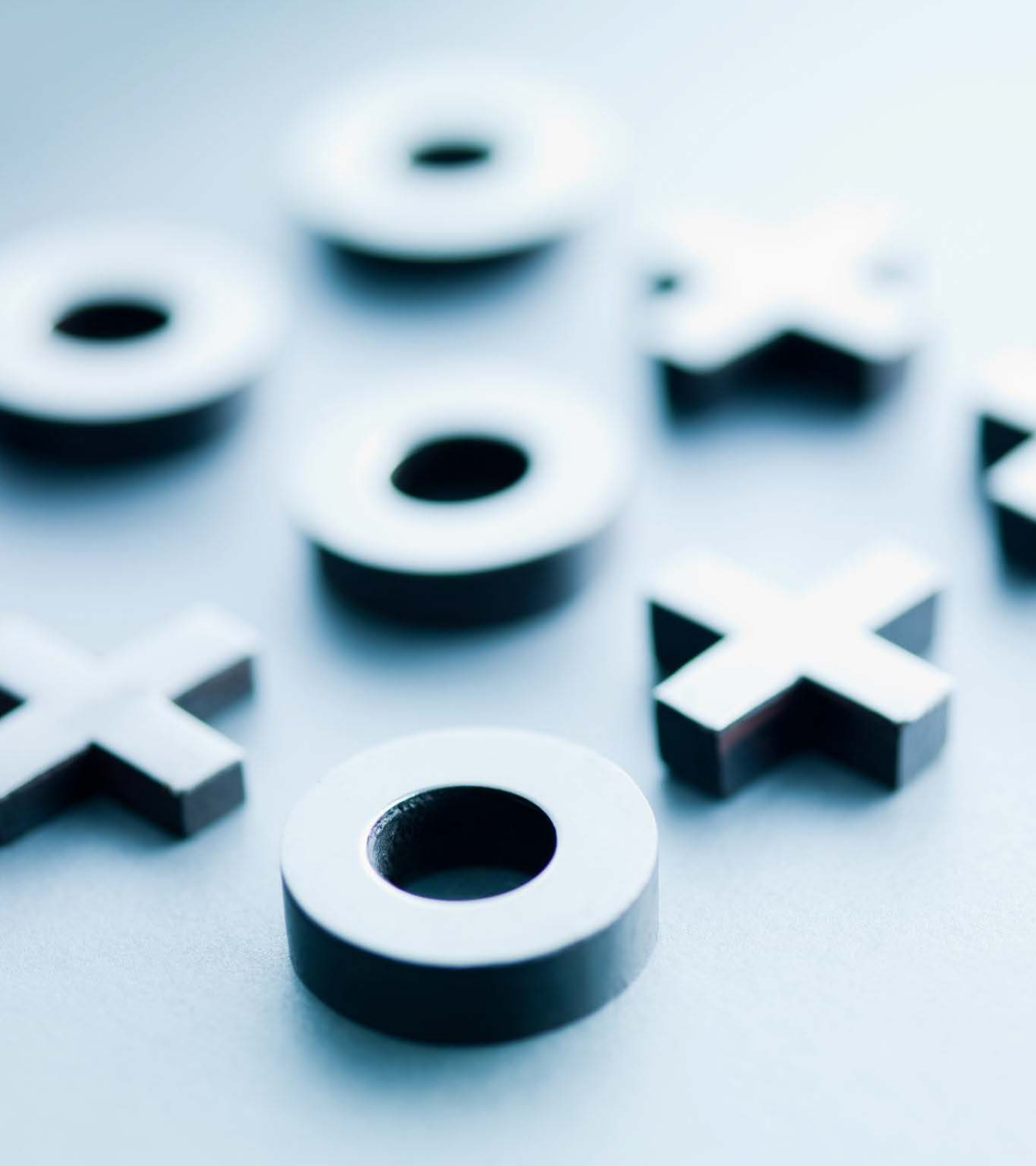


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# After experiment

- Flush gas lines containing dry/wet  $\text{NH}_3$
- Replace water in humidifier
- Replace scrubber solution.



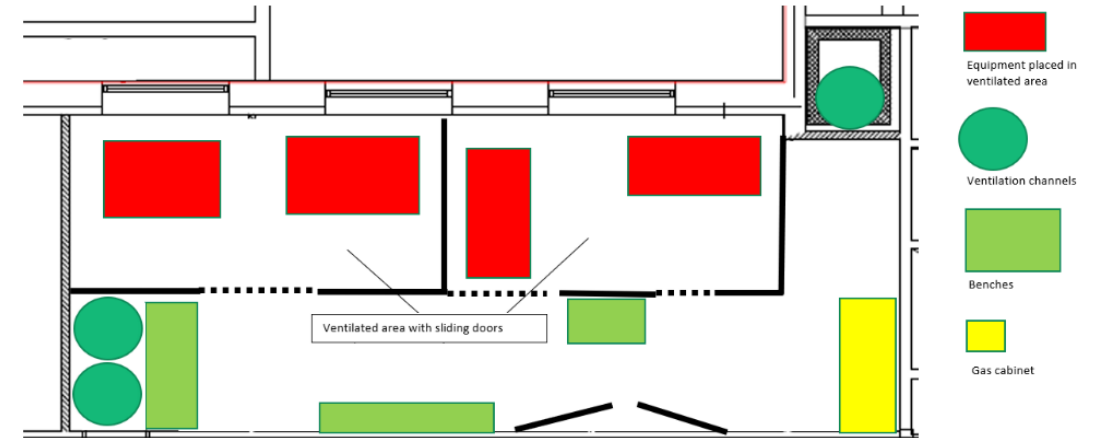


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# Testing infrastructure – NH<sub>3</sub>-lab

- Total area: ~60 m<sup>2</sup>
- Two ventilated areas separated from work space by sliding doors
- Ventilation capacity: 5000 m<sup>3</sup>/h
- 4 NH<sub>3</sub> sensors + sensors for LEL (H<sub>2</sub>) and CO
  
- Commissioned Jan 2023
- Shared with CoorsTek Membrane Sciences

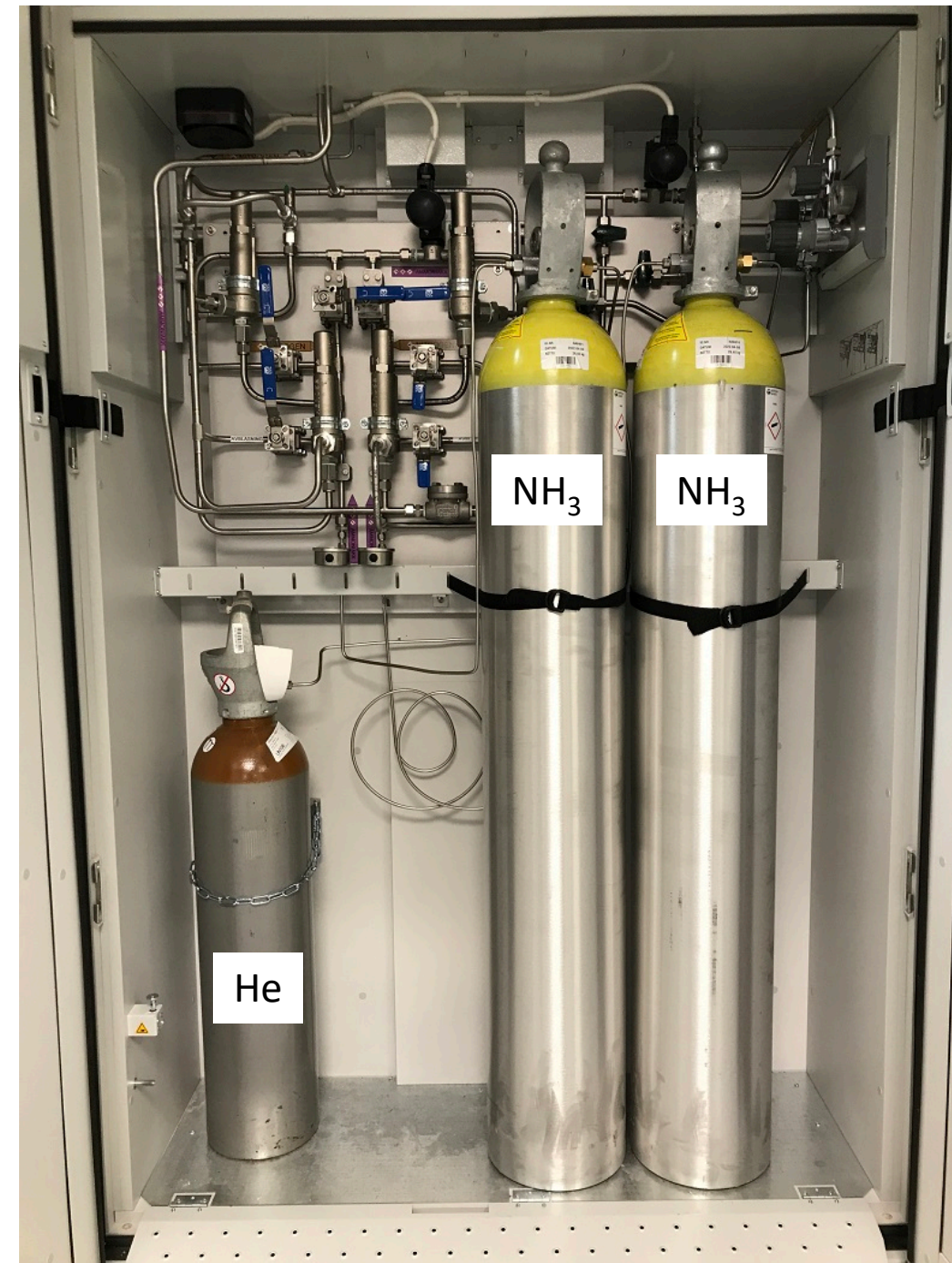




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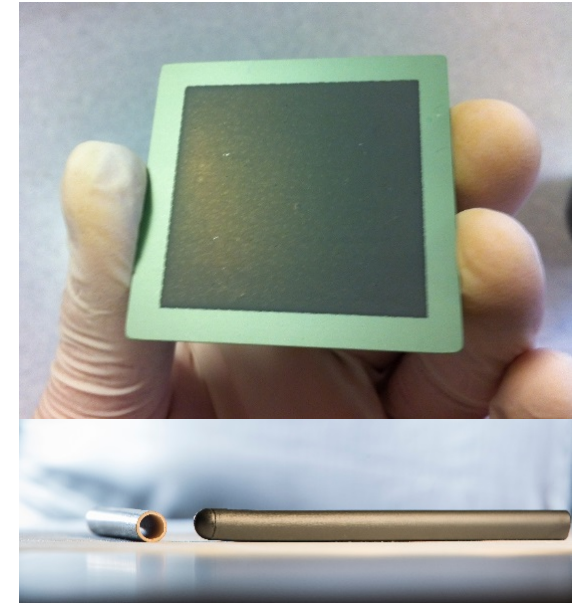
# NH<sub>3</sub>-lab

- Safety storage cabinet with 2 × 26 kg NH<sub>3</sub> (50 L)
- He for pressurizing the NH<sub>3</sub>
- N<sub>2</sub> for flushing of gas lines during bottle changes
- NH<sub>3</sub> sensor inside cabinet
- Magnetic solenoid valves connected to NH<sub>3</sub> line for automatic shut off in case warning level is exceeded



# Testing infrastructure – NH<sub>3</sub>-lab

- From small components and "button cells" to full-size planar (10x10 cm<sup>2</sup>) and tubular fuel cells and short stacks (5 kW)
- Infrastructure for H<sub>2</sub>, O<sub>2</sub>, air, CO, CO<sub>2</sub>, CH<sub>4</sub>, He, Ar, H<sub>2</sub>S and NH<sub>3</sub>
- Micro-GC, MS and FTIR for gas analysis
- New lab dedicated to NH<sub>3</sub> activities
  - Current: Greenlight X40 station for single cell testing
  - Planned: corrosion furnace, Pd testing setup, ProboStat testing setup



# Concluding remarks

## High temperature experiments with $\text{NH}_3$

### Prior to experimental start-up

- Follow all HSE procedures! Concentrated  $\text{NH}_3$  is deadly.
- Replace all valves, gaskets, MFCs
- Careful evaluation of gas-inlet materials.

### During experiment

- Wet  $\text{NH}_3$  is highly corrosive
- Expect  $\text{Ni}_3\text{N}$  formation

### After experiment

- Flush lines, clean scrubbers





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