Vacuum measurements of materials and coatings for GWD beampipes

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OUTLINE

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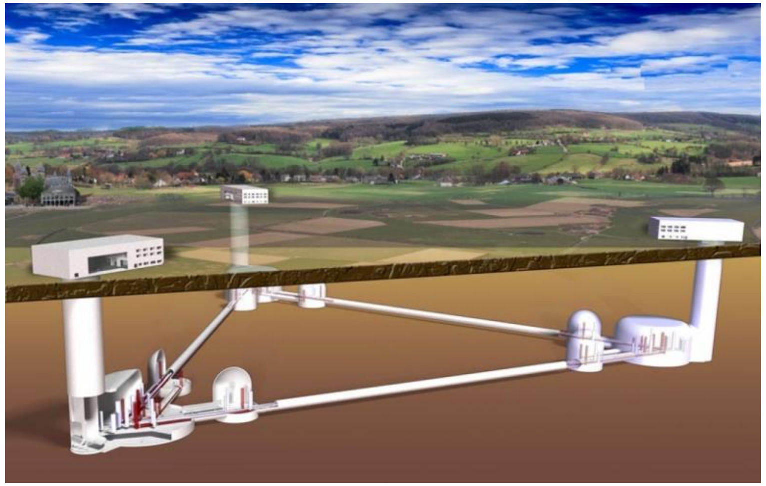
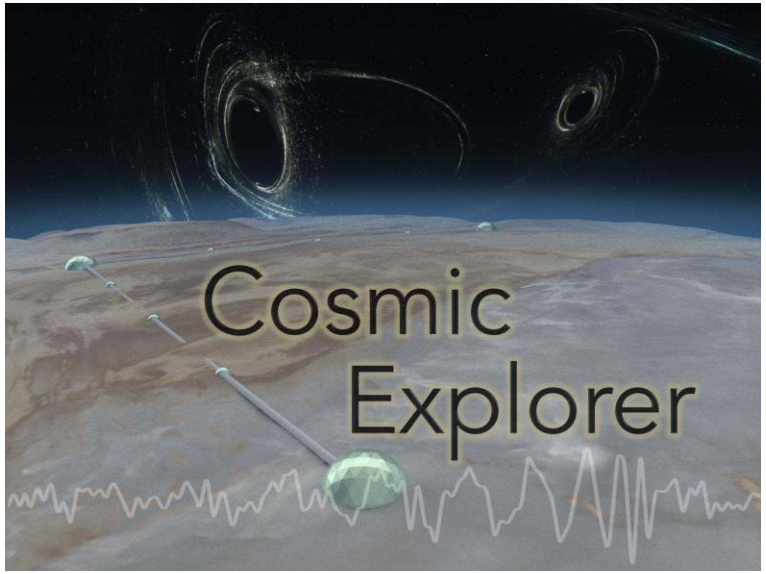
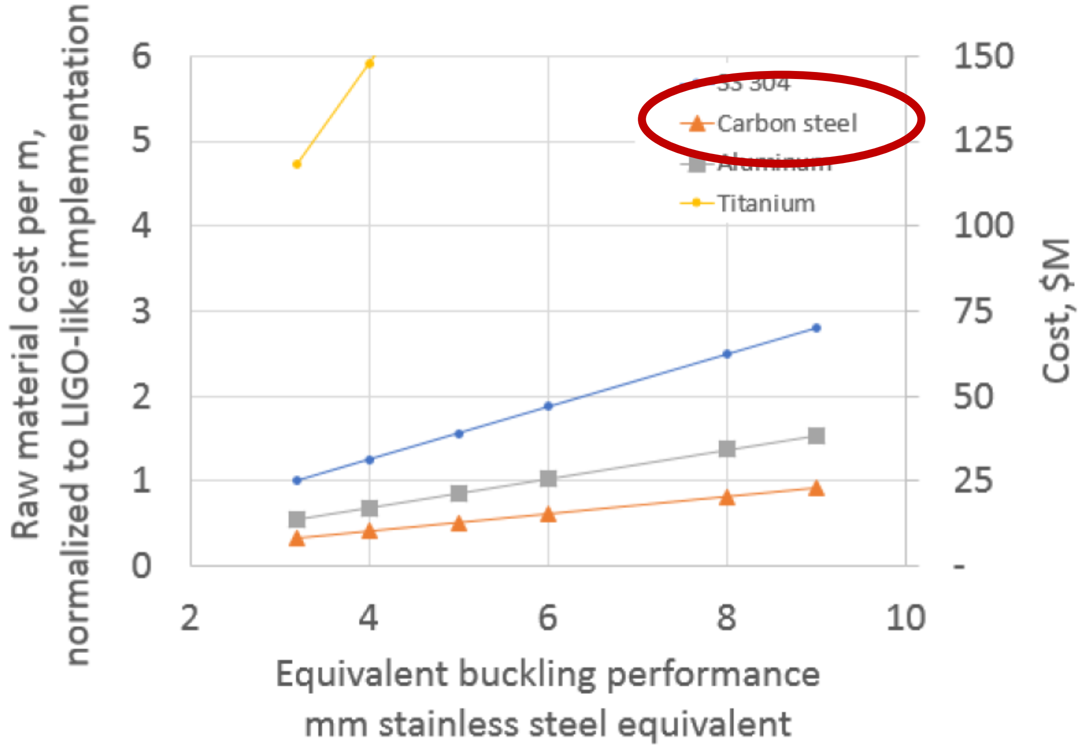
- MILD STEEL
  - ✓ Samples
  - ✓ Water outgassing
  - ✓ Hydrogen content
  - ✓ Hydrogen outgassing
  - ✓ Total outgassing
- SILICON COATING
  - ✓ Samples
  - ✓ Water outgassing
  - ✓ Hydrogen content
  - ✓ Hydrogen outgassing
- CONCLUSIONS





Cost reduction guidelines

Beamtube material



Bake-out

Lower bake-out temperatures for longer durations

Beamtube coatings with low water vapor binding energy:

magnetite Fe_3O_4

silicon

NSF Workshop on Large Ultrahigh-Vacuum Systems for Frontier Scientific Research Instrumentation, LIGO-P1900072-v1, H.F Dylla, R. Weiss and M.E. Zucker, eds

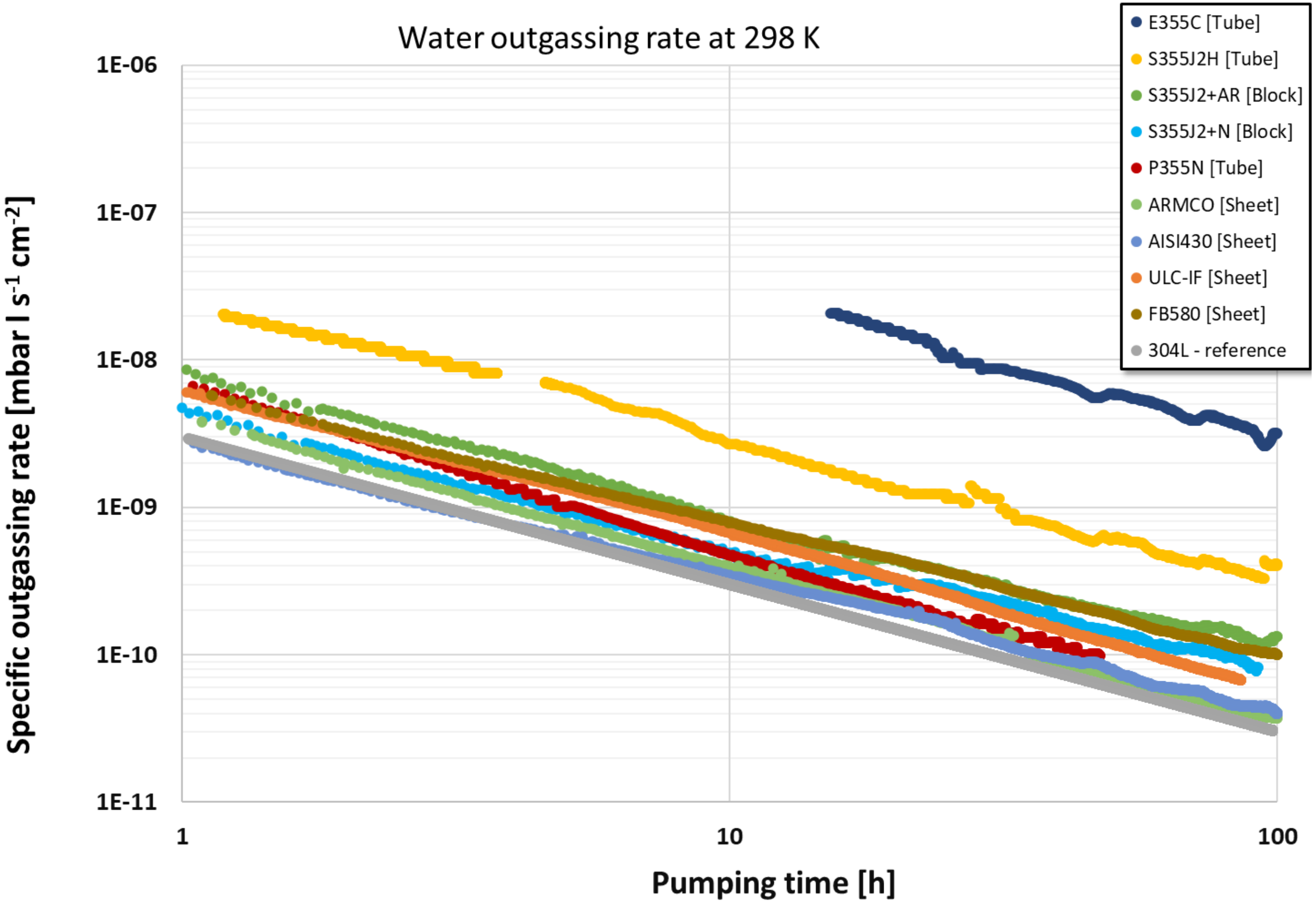
Mild Steel

Materials & Methods

Class	Grade	Shape
Ferritic/pearlitic	P355	Tube
	E355	Tube
	S355J2H	Tube
	S355J2+N	Block
	S355J2+AR	Block
Ferritic/bainitic	FB580	Sheet
Ferritic	ARMCO	Sheet
	ULC-IF	Sheet
Ferritic (SS)	AISI430 BA	Sheet

Method	Result
TDS	H content
Pumpdown	H_2O outgassing
Coupled method	H_2, CH_4, CO & CO_2 outgassing
Binding energy	H_2O binding energy

Mild steel: Water outgassing

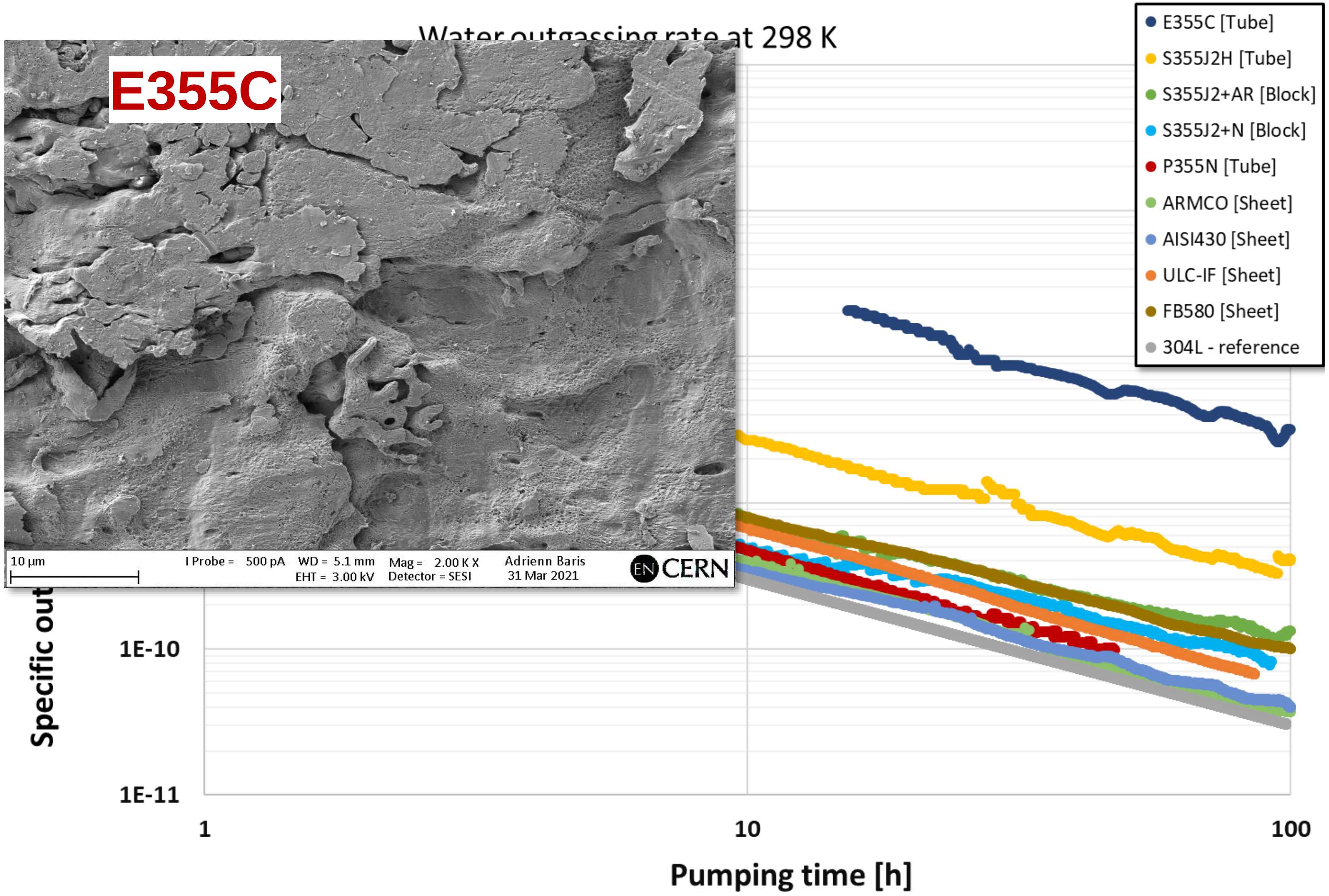


Sample	Q_{10h} [mbar l s^{-1} cm^{-2}]
E355C	$3.0\ 10^{-8}$**
S355J2H	$2.7\ 10^{-9}$
P355N	$4.7\ 10^{-10}$
S355J2+AR	$8.0\ 10^{-10}$
S355J2+N	$4.9\ 10^{-10}$
FB580	$7.9\ 10^{-10}$
ULC-IF	$6.7\ 10^{-10}$
ARMCO	$3.9\ 10^{-10}$
AISI430 BA	$3.5\ 10^{-10}$
304L	$3.0\ 10^{-10}$[1]

Data contribution by A. Michet & N. Thaus

1 Chiggiato P., Outgassing properties of vacuum materials for particle accelerators. arXiv. 2020. https://doi.org/10.48550/arXiv.2006.07124

Mild steel: Water outgassing



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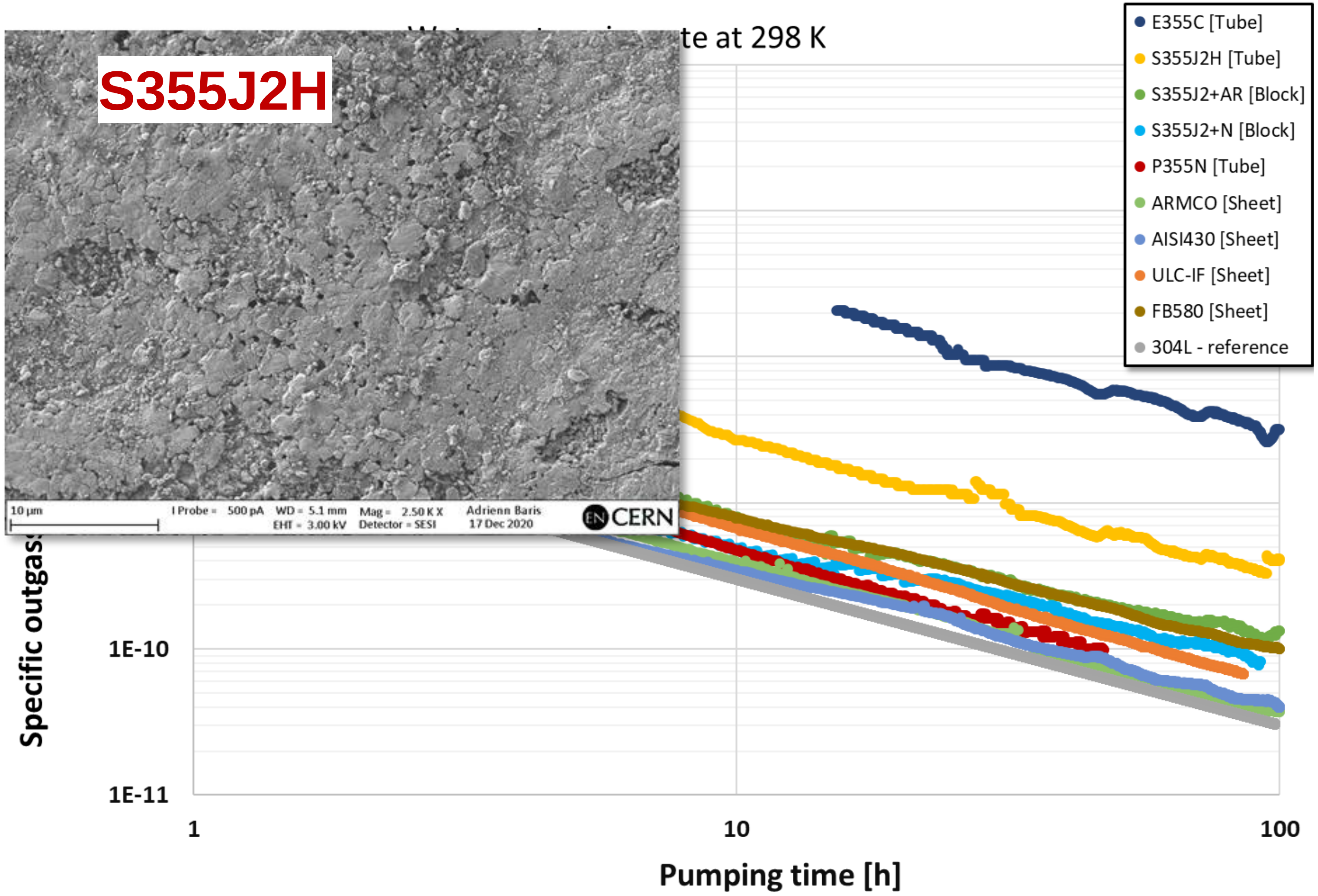

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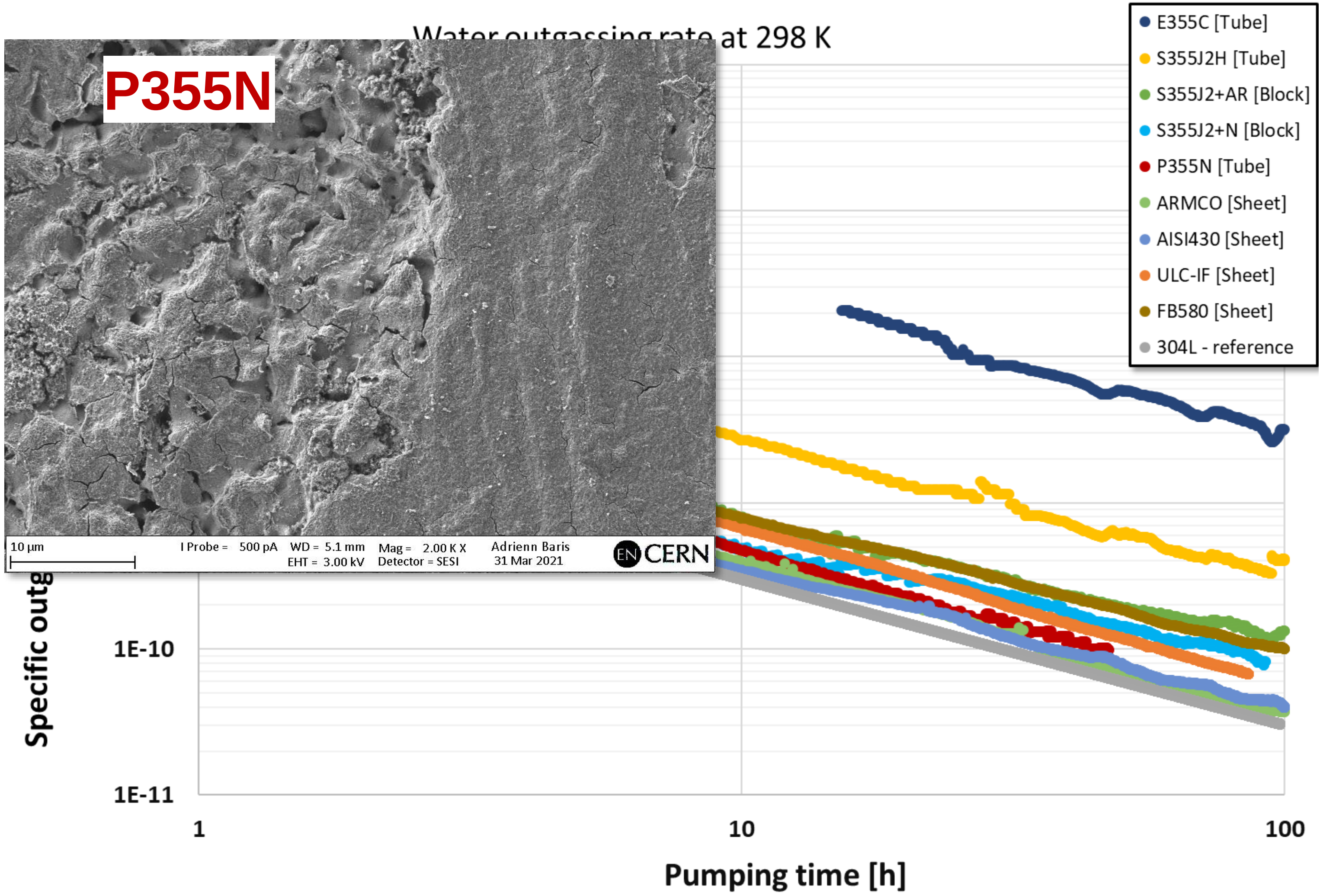

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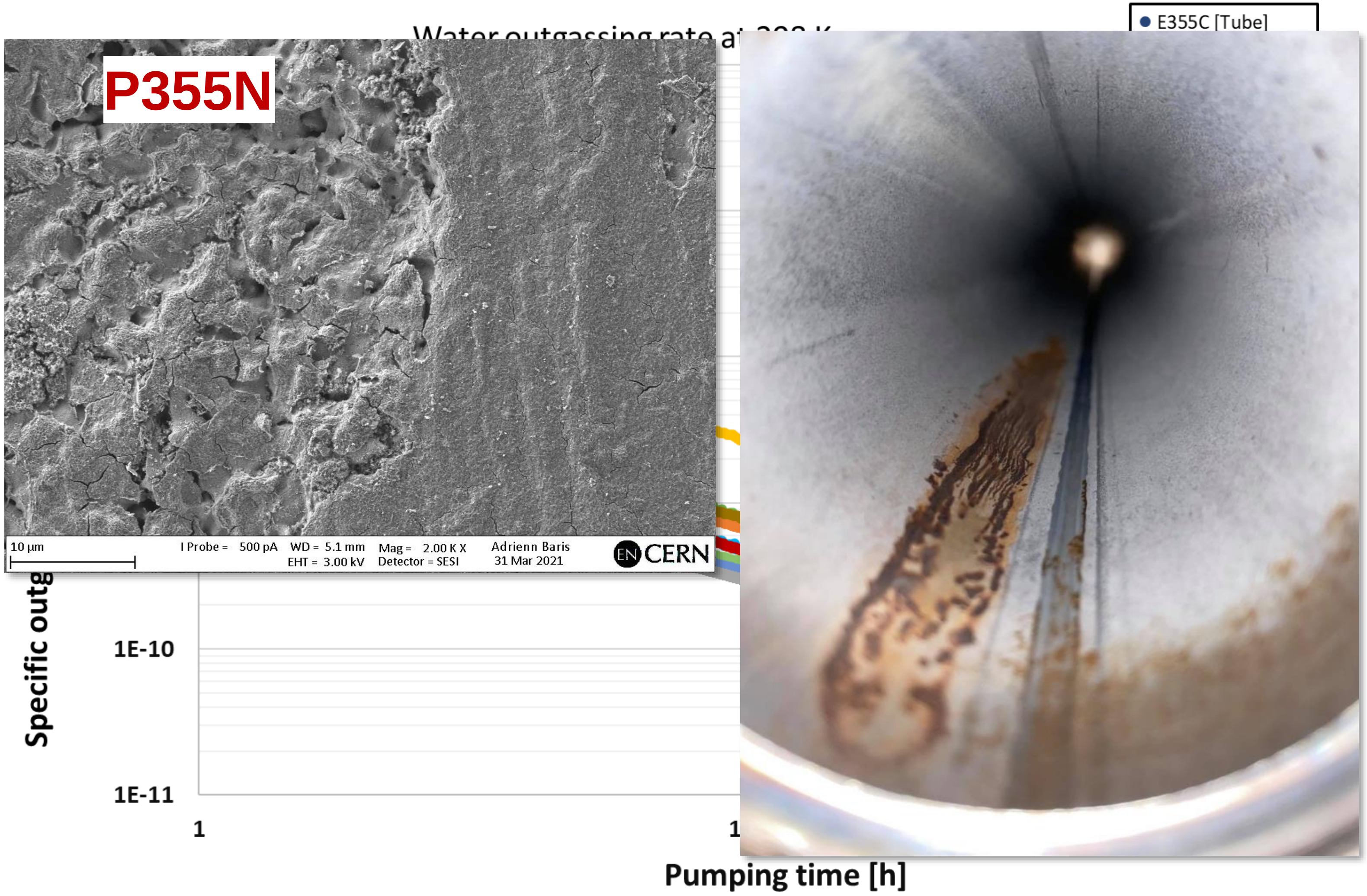

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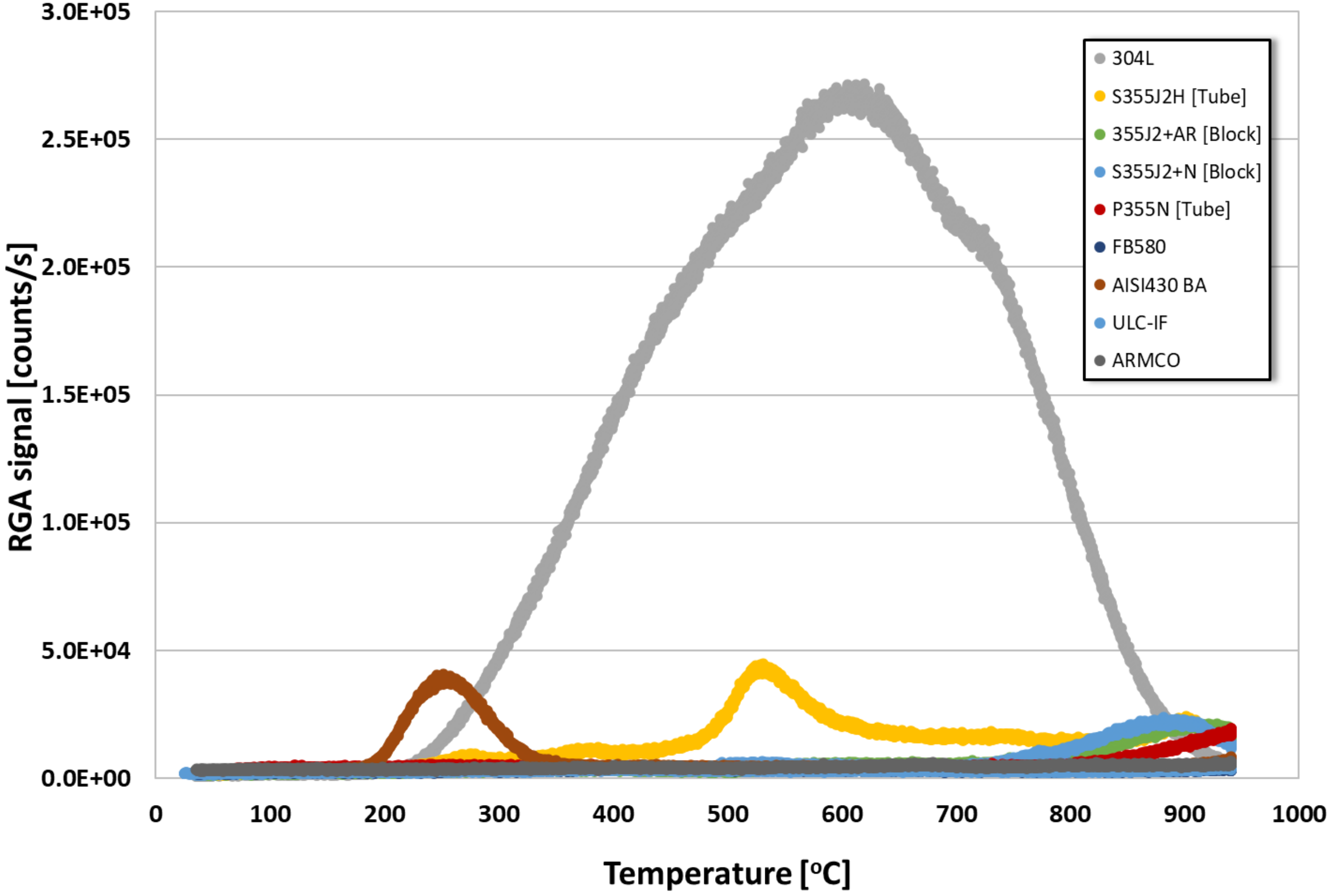
Mild steel: Thermal desorption

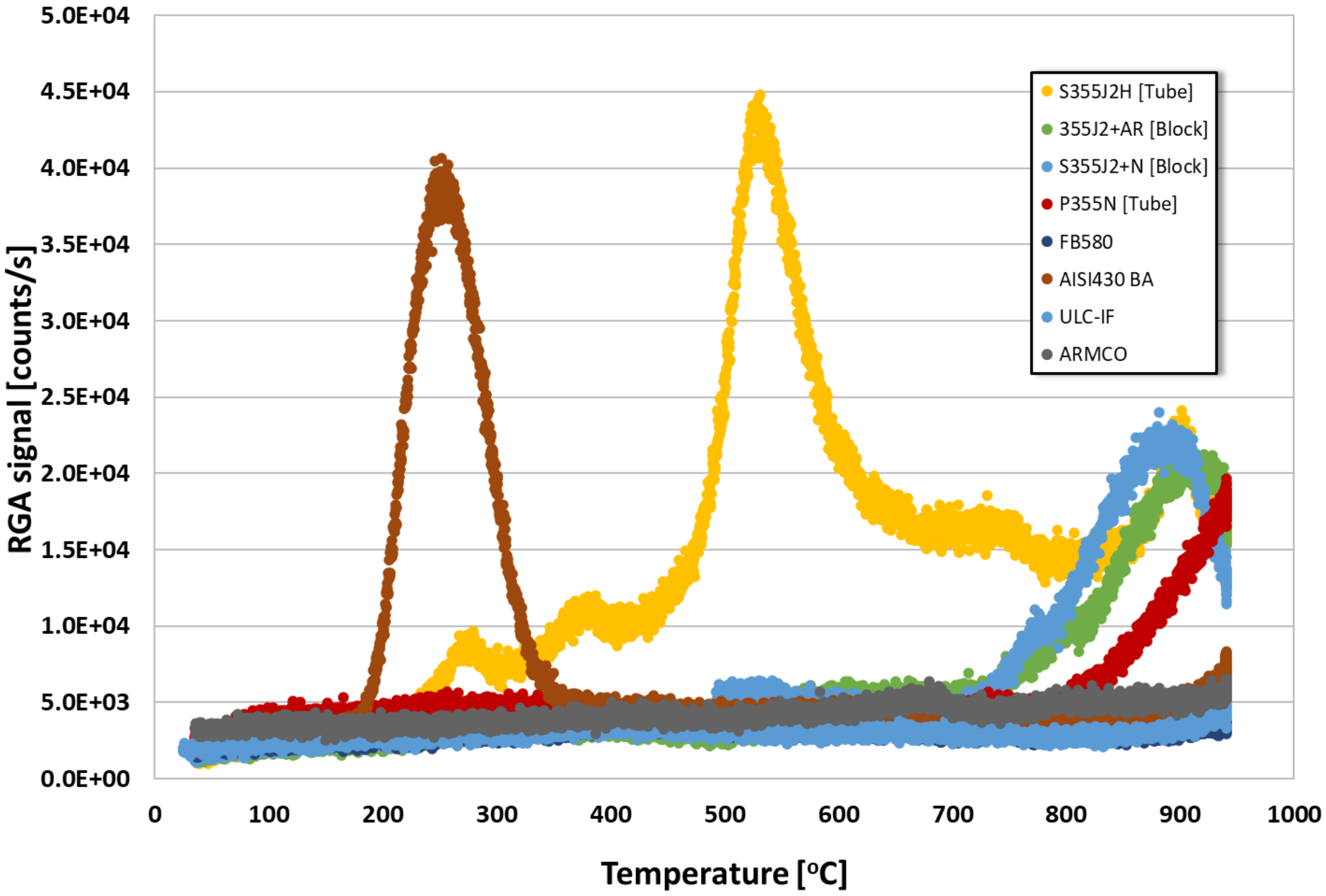


Sample	Released H_2 [ppm at.]	Thickness [mm]
304L	80	3
S355J2H	7.8	4
S355J2+AR	2.0	3
S355J2+N	1.6	3
P355N	1.0	3.5
FB580	2.8	2.1
AISI430 BA	8.3	2
ULC-IF	3.7	0.69
ARMCO	1.2	2

Data contribution by A. Michet

*Quantities of released hydrogen are calculated up to 850°C

Mild steel: Thermal desorption



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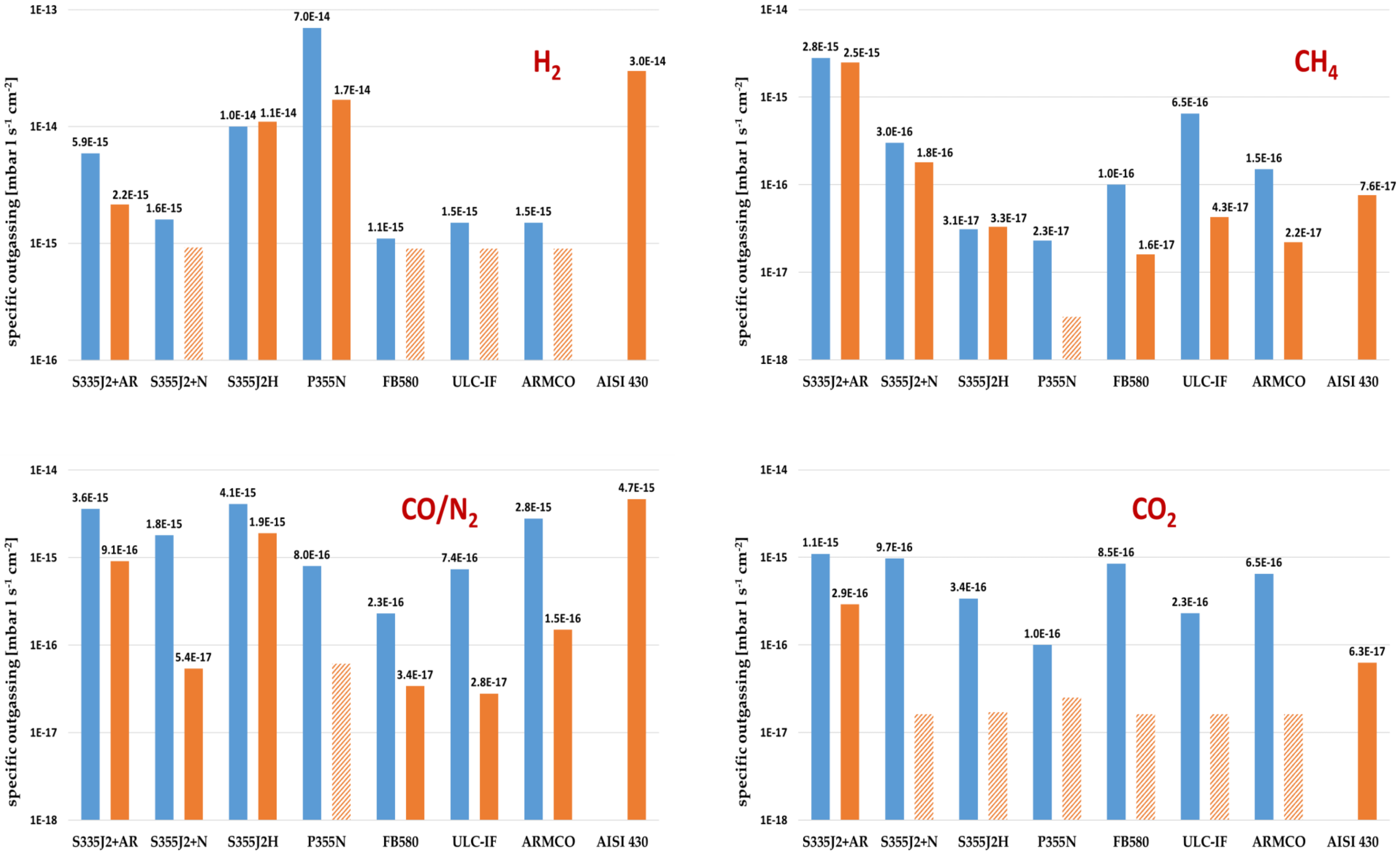
Outgassing – coupled method

80C/48h

150C/48h

Detection limit



Detection limit

50% of background

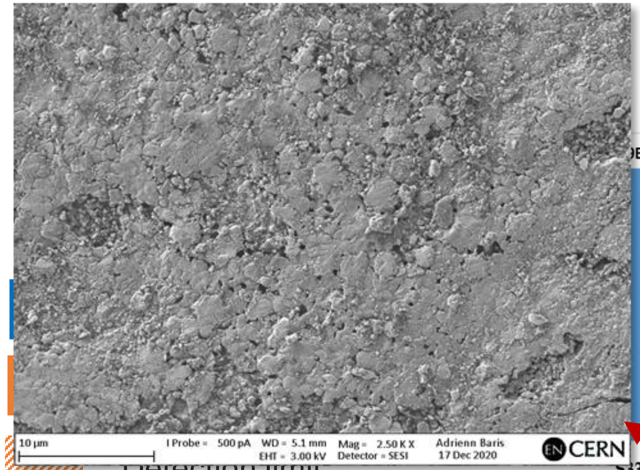
Measurement error

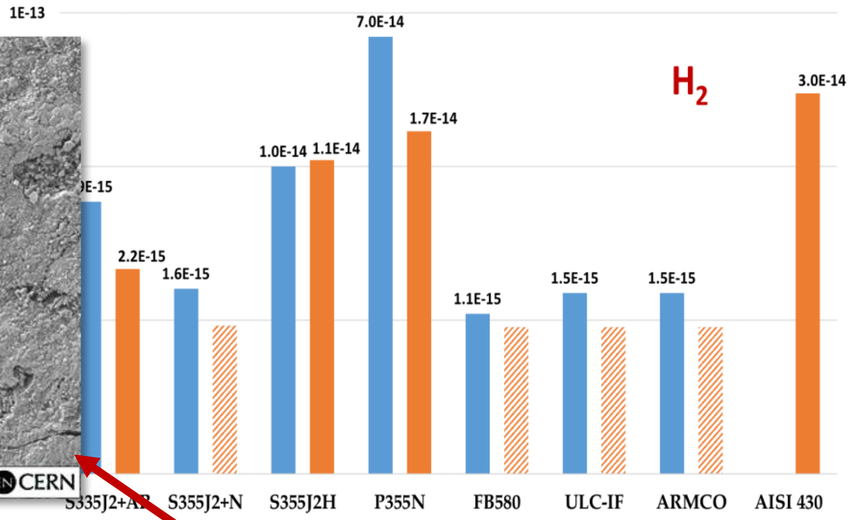
40%

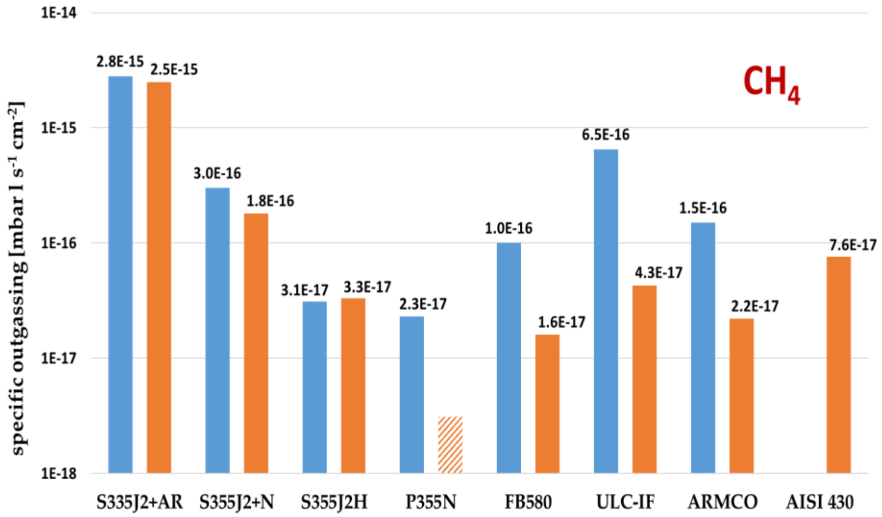
Mild steel vacuum chamber

- Mild steel tubes
- Welded 316LN vacuum fired flanges

Outgassing – coupled method







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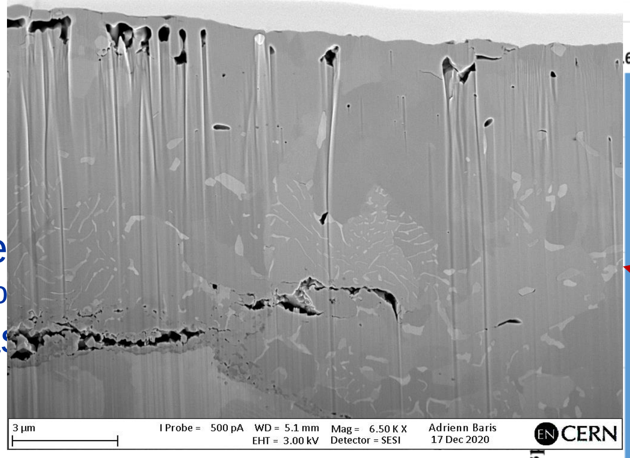
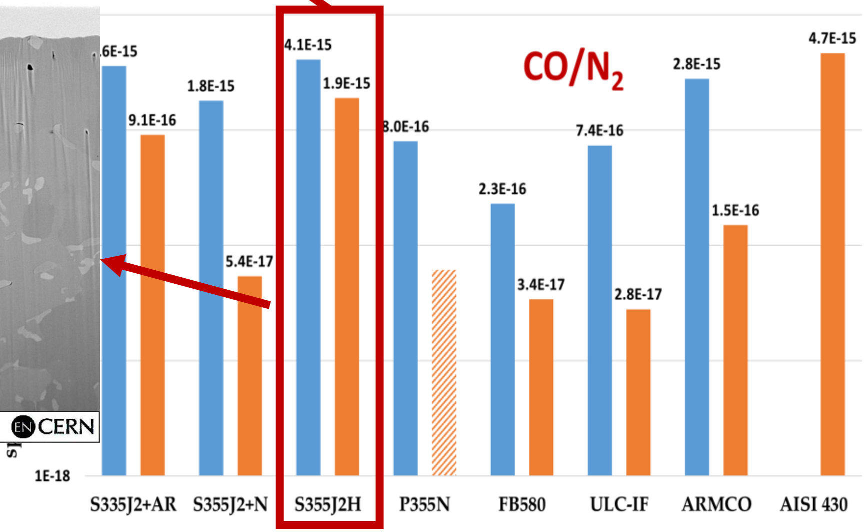
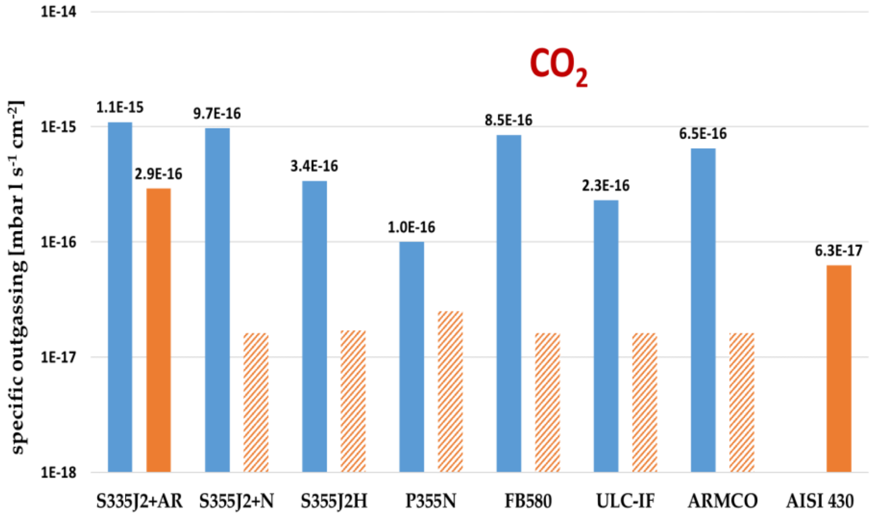






Dete

50% o

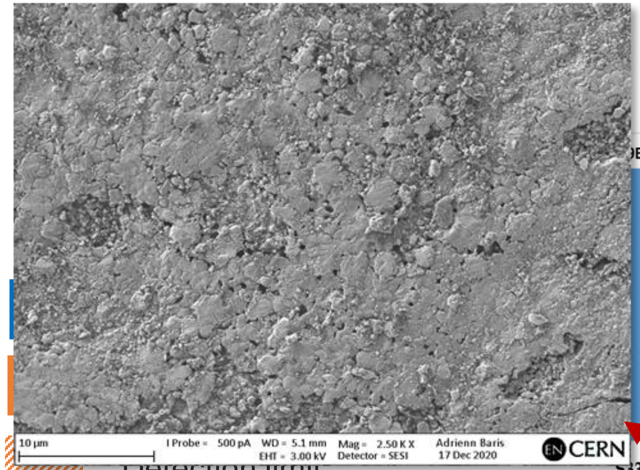
Meas

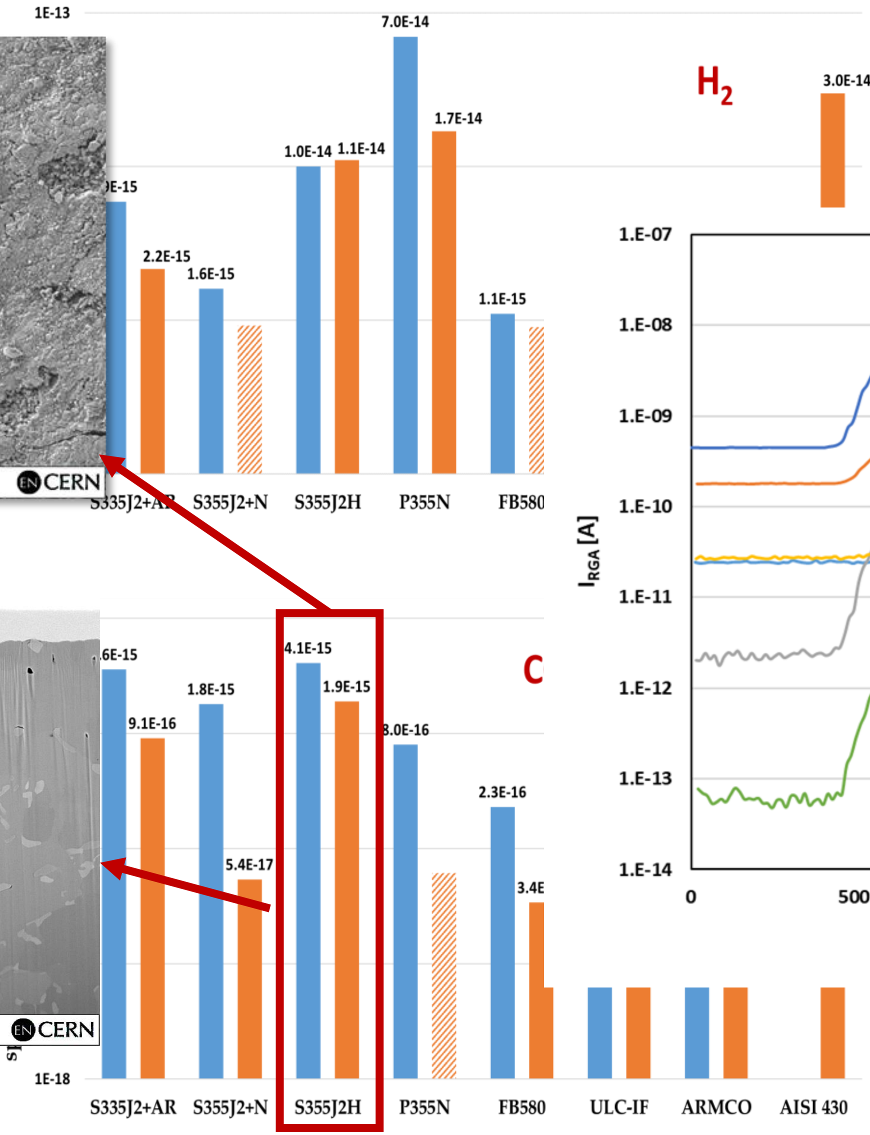
40%

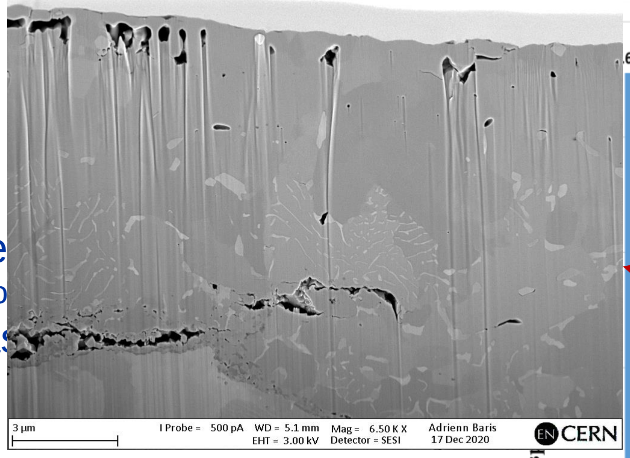
Mild

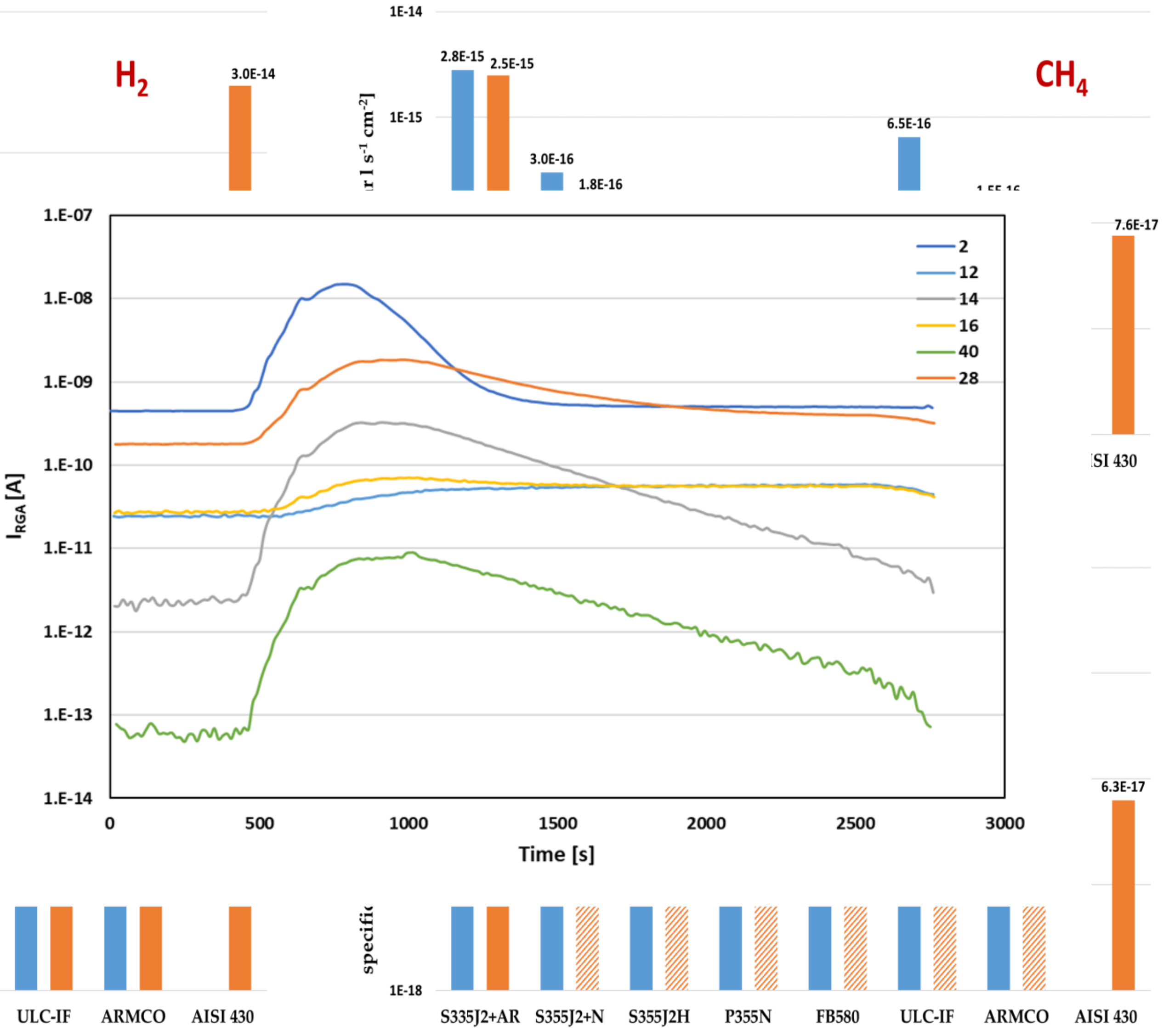
- Mild steel tubes
- Welded 316LN vacuum fired flanges

Outgassing – coupled method









Dete...
50% o...
Meas...
40%
Mild...

- Mild steel tubes
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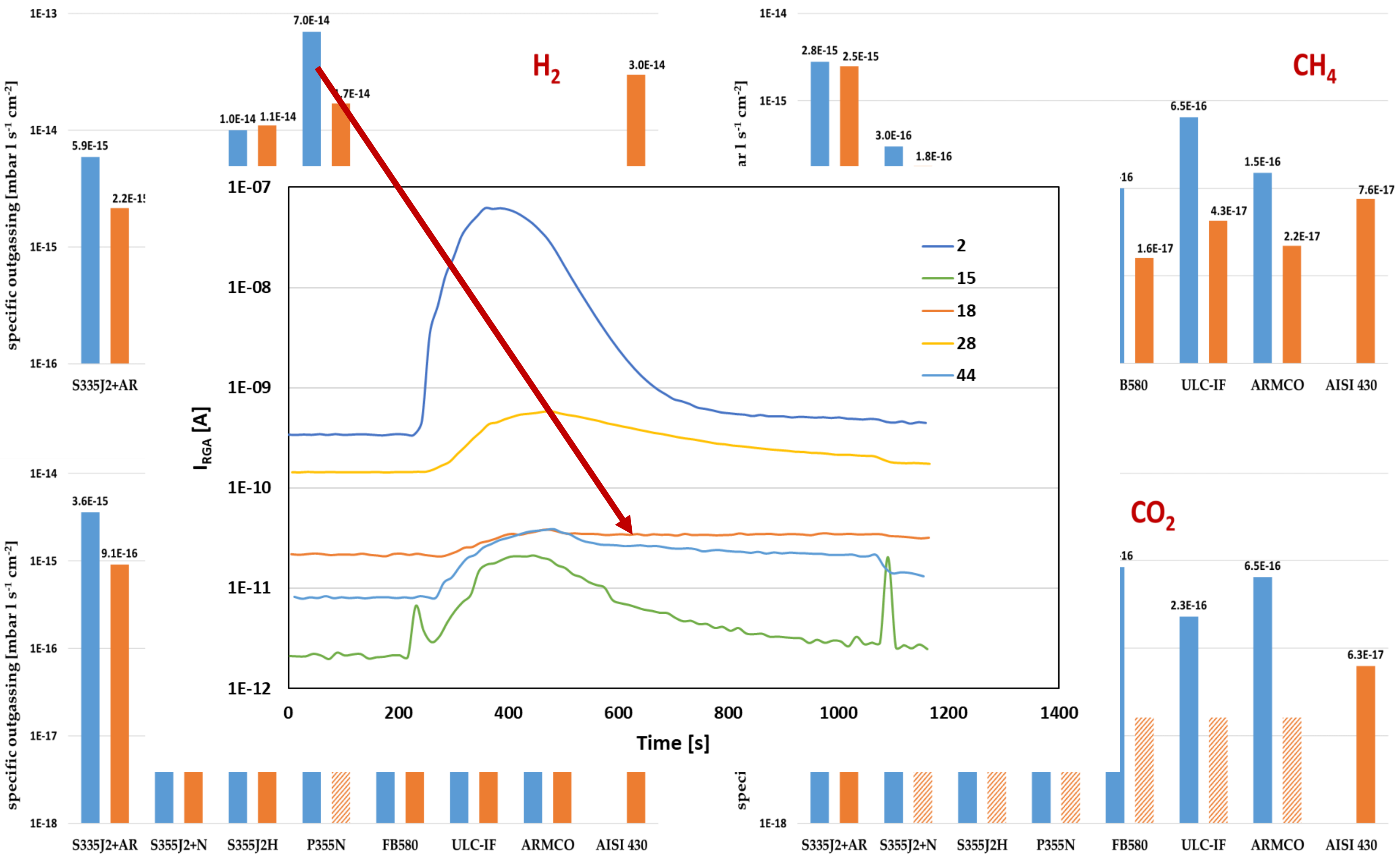
Outgassing – coupled method

80C/48h
150C/48h
Detection limit



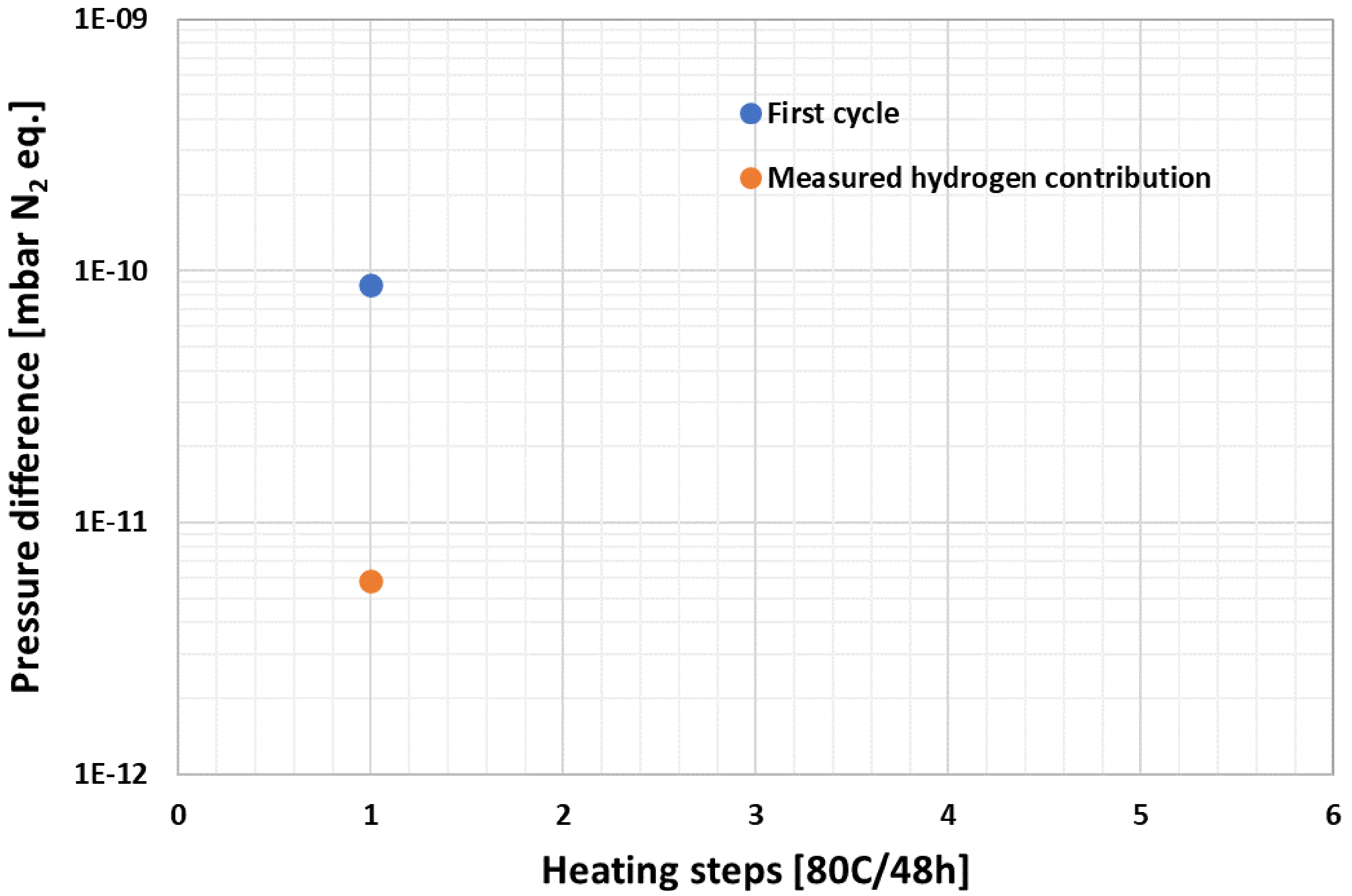
Detection limit
50% of background

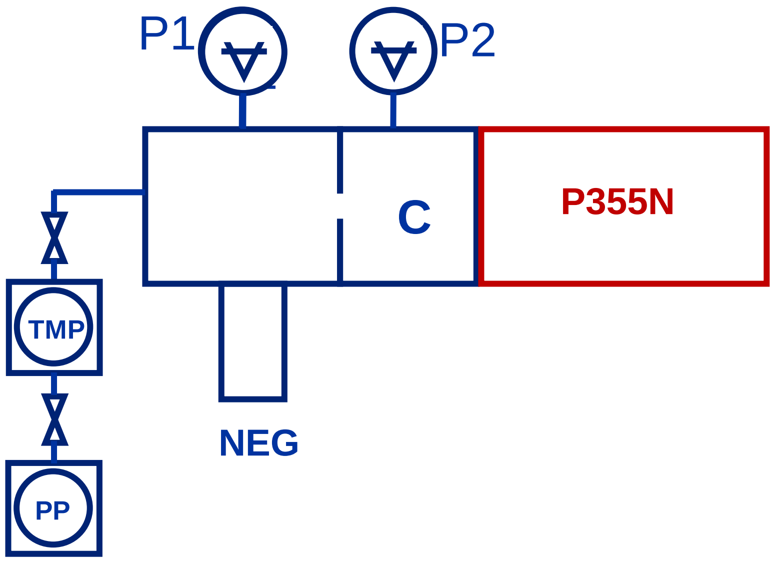
Measurement error
40%

Mild steel vacuum chamber

- Mild steel tubes
- Welded 316LN vacuum fired flanges

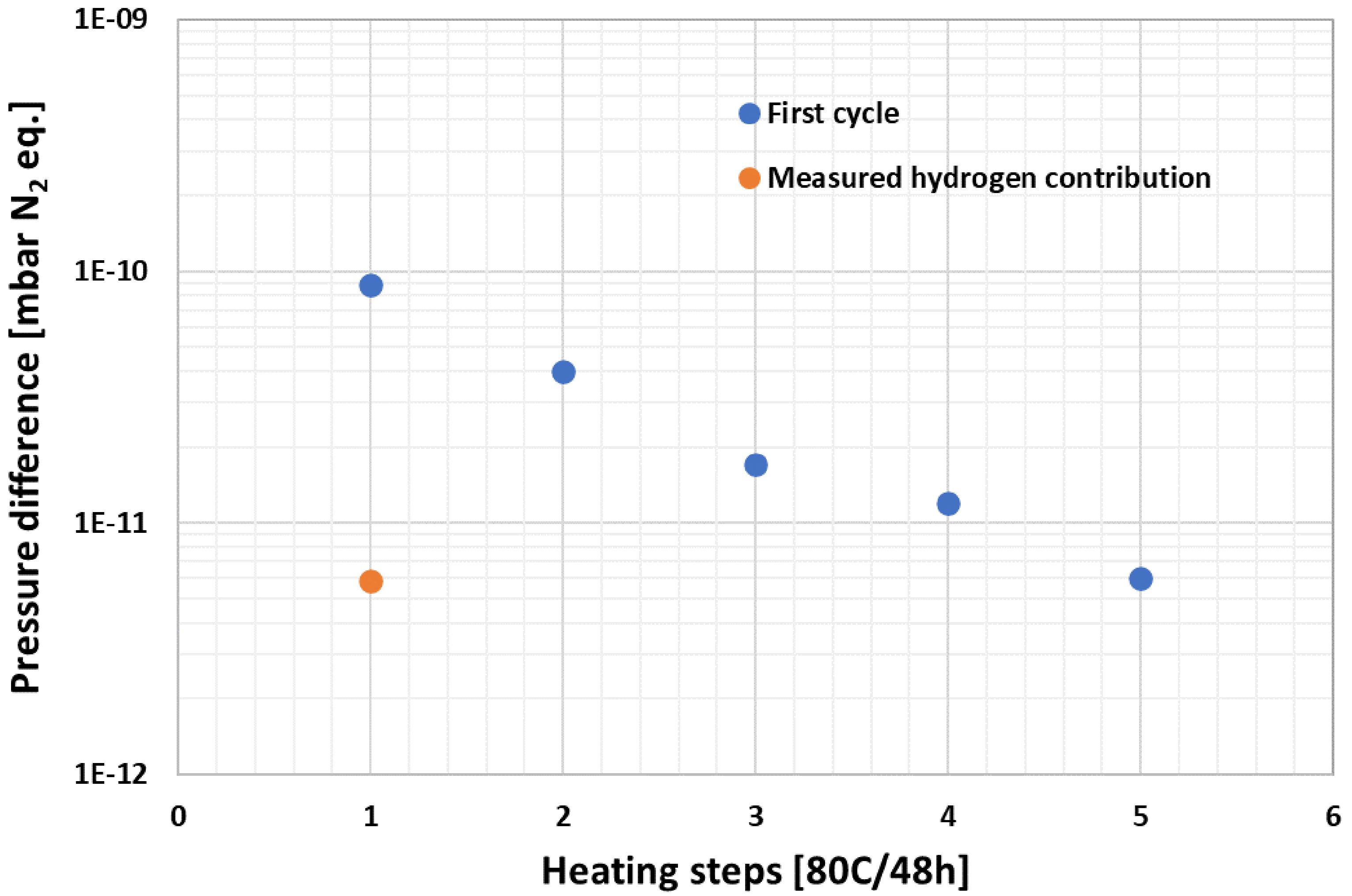
Ultimate pressure – P355N

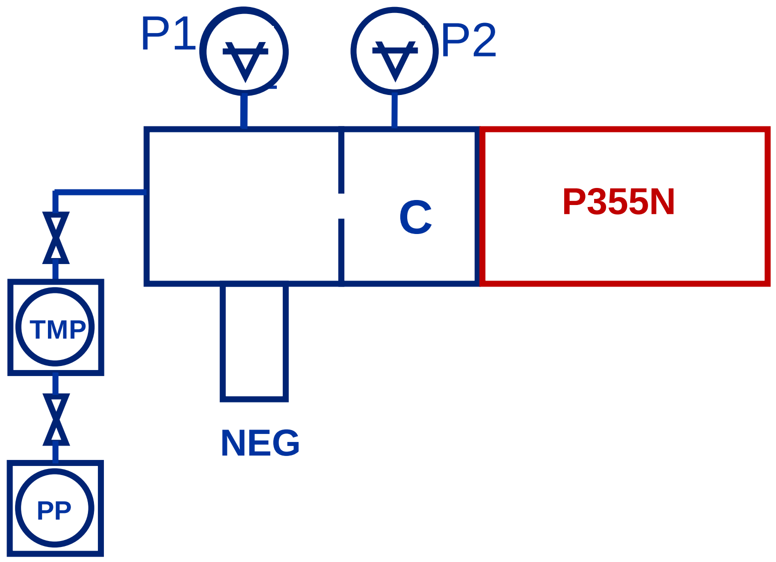




- Throughput system: ø 10 mm orifice – Surface area P355N chamber = 6333 cm^2
- Background pressure removed
- The hydrogen outgassing rate of an as-received chamber after heating 80ºC for 48 hours is 7 10^{-14} mbar l s^{-1} cm^{-2} . The outgassing rates of the other gasses (CH_4, CO/N_2 and CO_2) are at least 10x lower

Ultimate pressure – P355N





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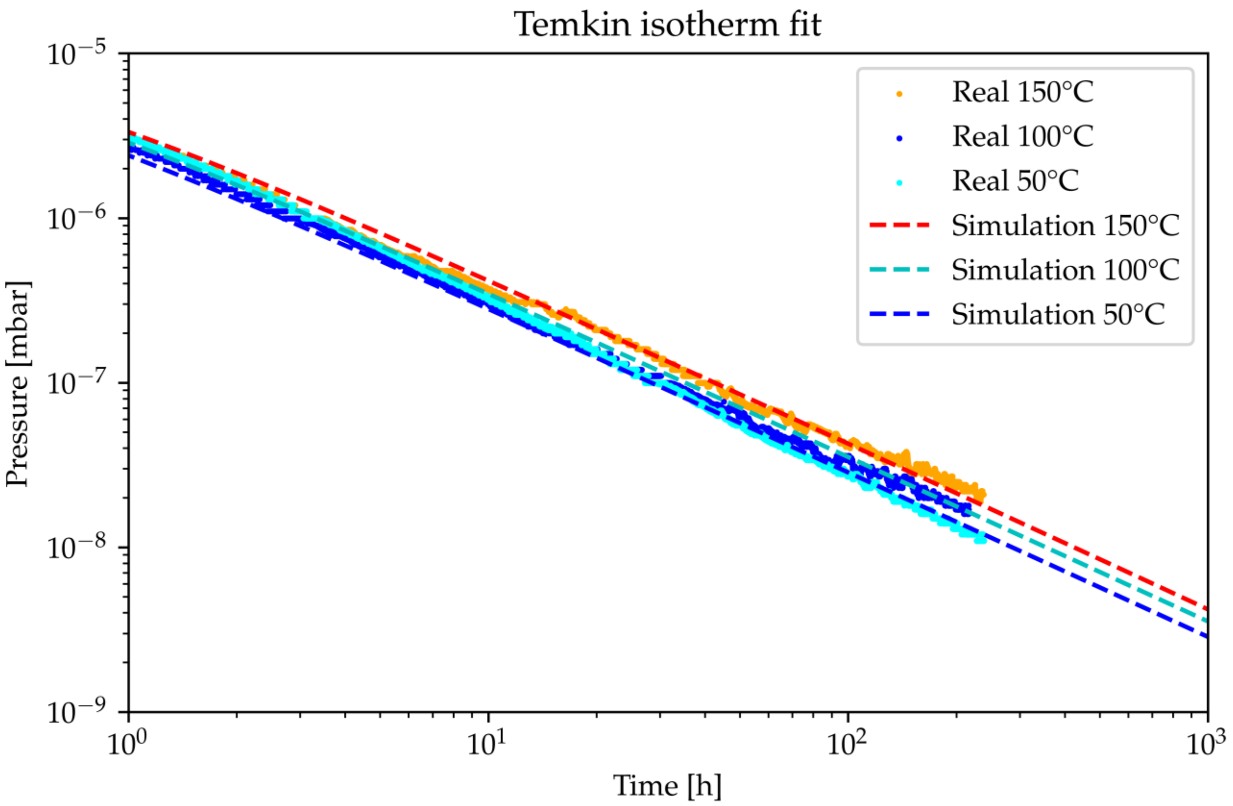
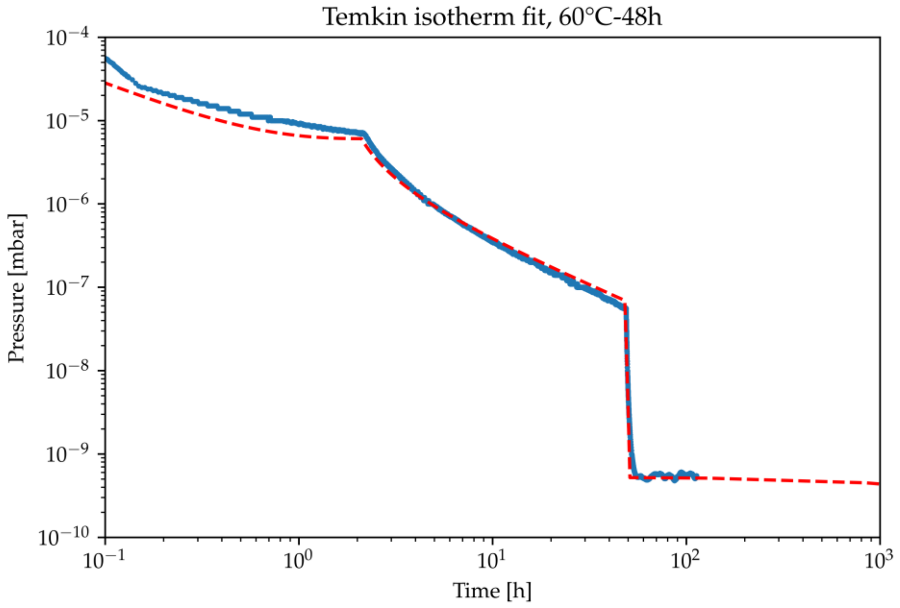
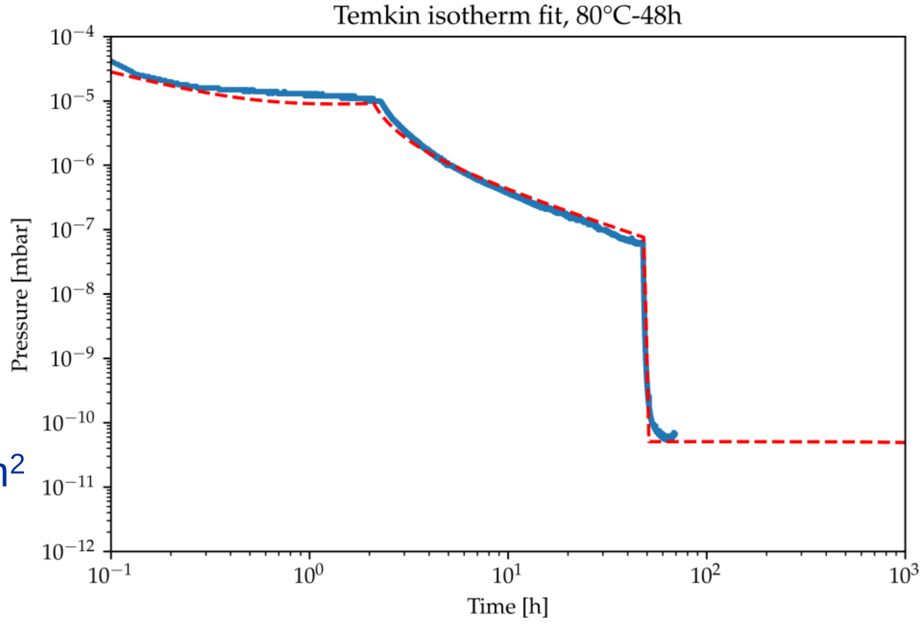
Water binding energy – P355N



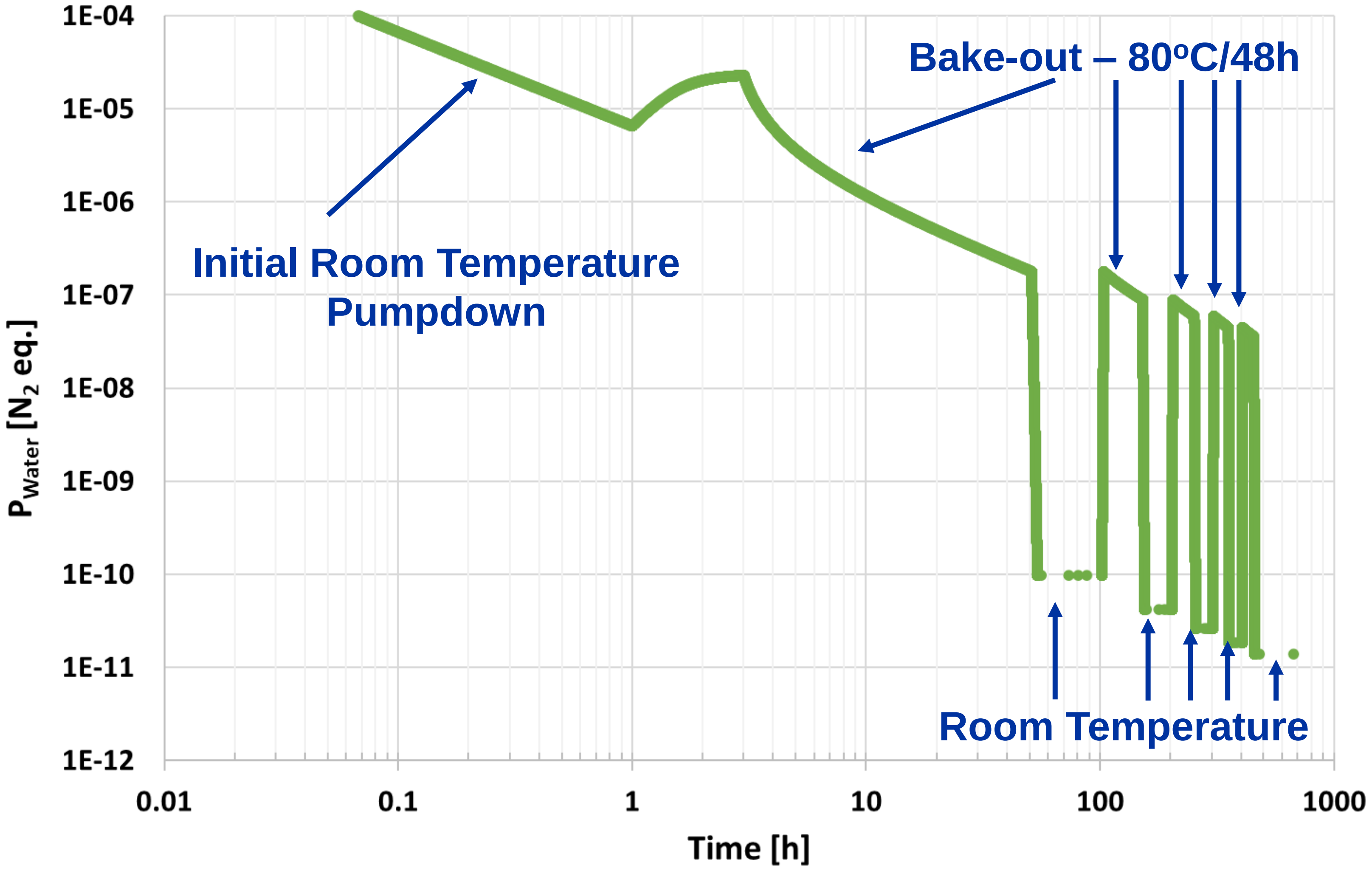
n_m
E_0, E_1





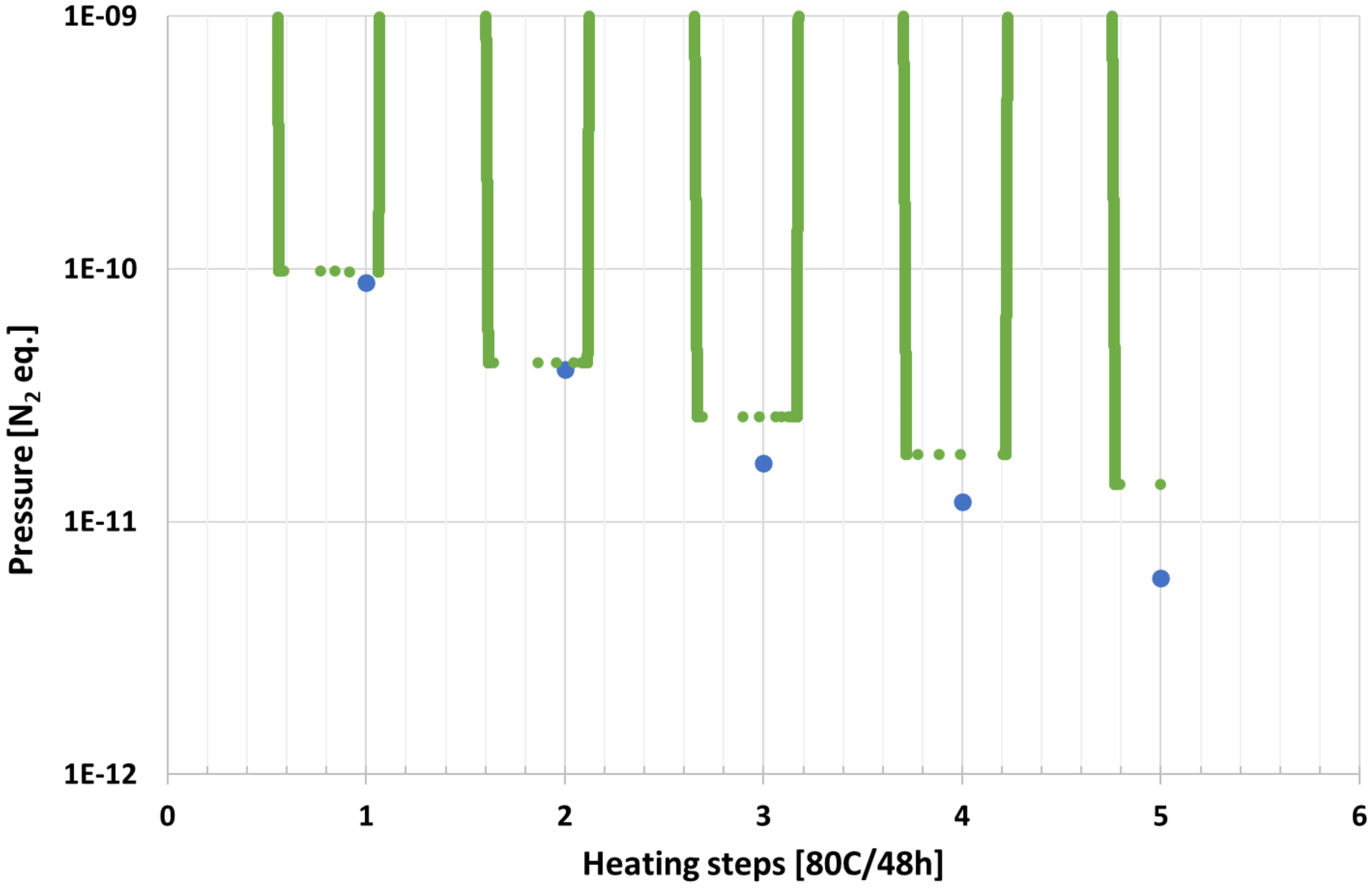
n_m : number of sites (molecules) per unit of surface	4.5 10^{20} molecules/cm^2
E_0 : Binding energy at Theta = 0 (zero coverage)	1.5 eV
E_1 : Binding energy at Theta = 1 (maximum coverage)	0.32 eV

Ultimate pressure – P355N



- Simulation of water pressure with 5 heating steps at 80ºC for 48 hours
- The number of available sites has been kept constant

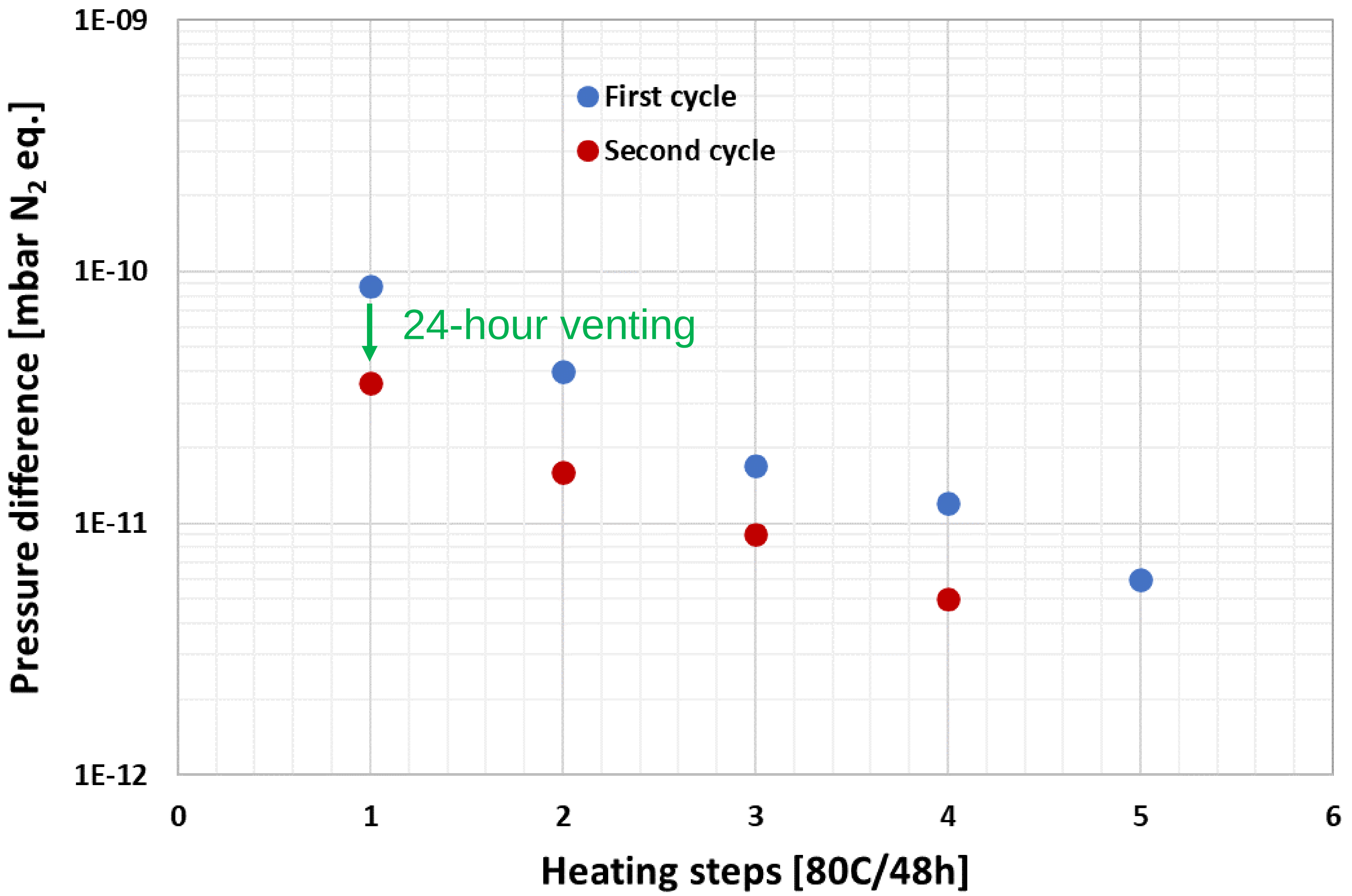
Ultimate pressure – P355N

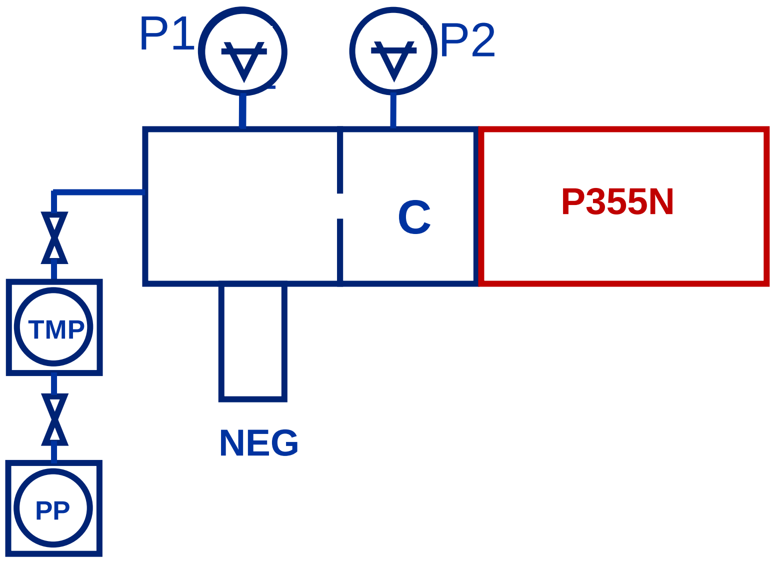


- Pressure ($P_2 – P_1$) nitrogen eq. in function of heating steps at 80ºC for 48 hours
- Simulation of water pressure with 5 heating steps at 80ºC for 48 hours
- The number of available site has been kept constant
- Discrepancy between measurement and simulation due to:

  -Number of available sites ≠ constant ?

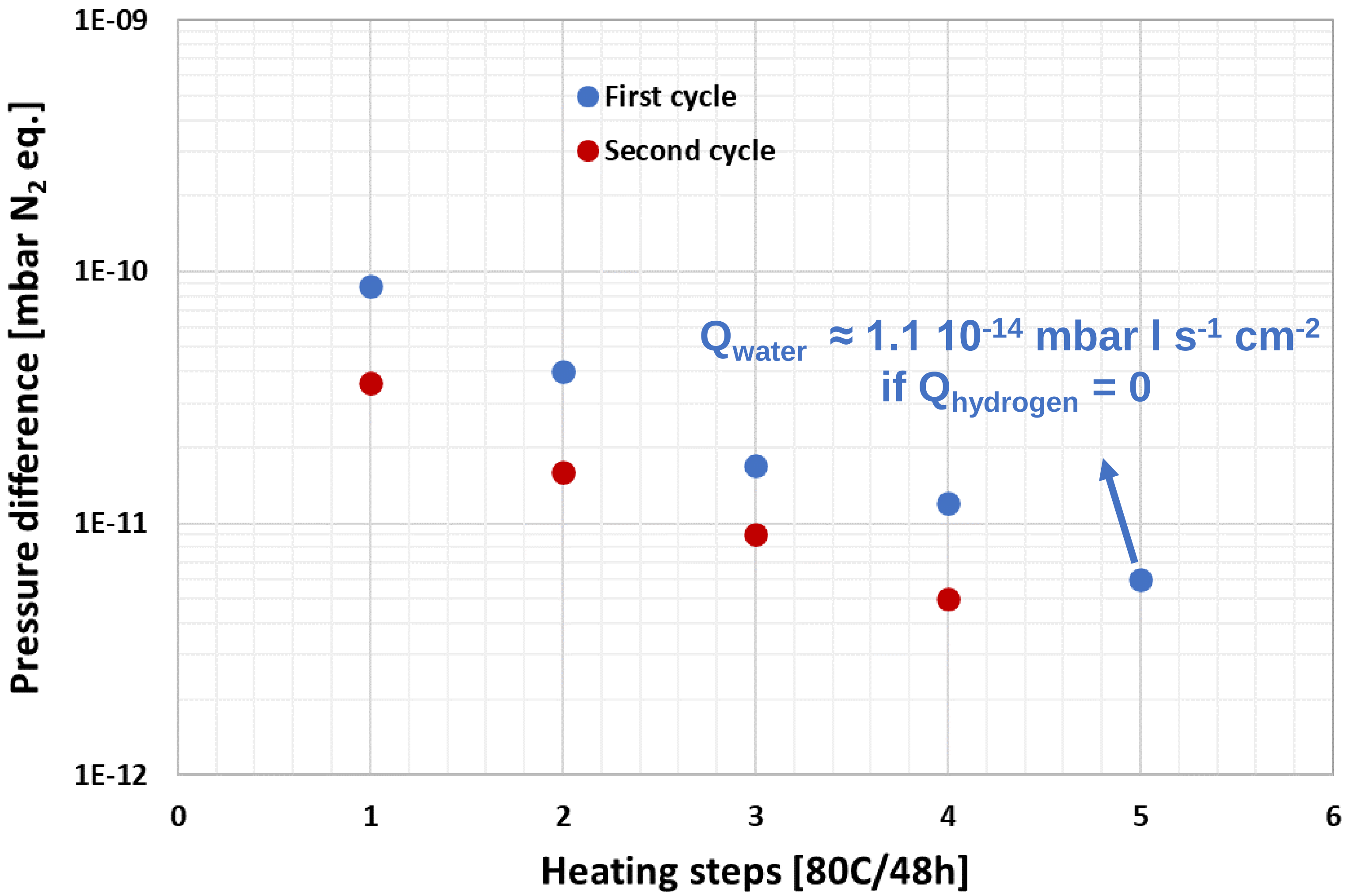
Ultimate pressure – P355N

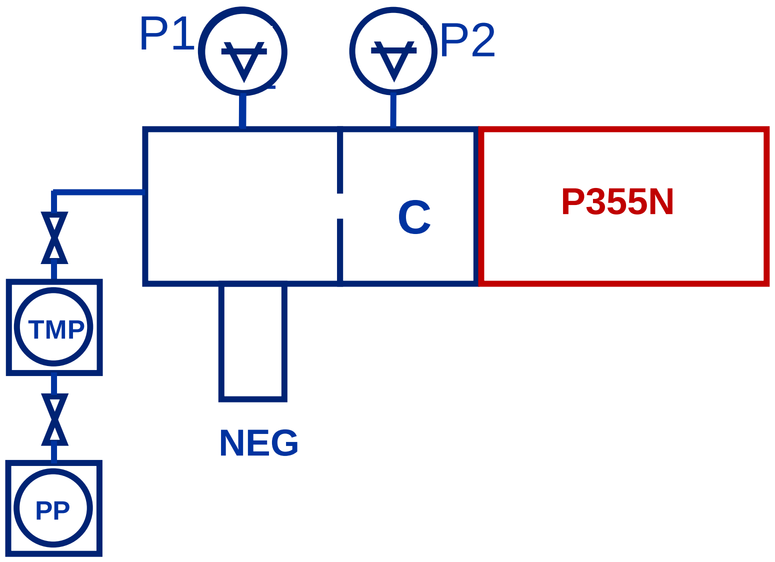




- Throughput system: ø 10 mm orifice – Surface area P355N chamber = 6333 cm^2
- Background pressure removed
- Pressure drop after 24-hour venting

Ultimate pressure – P355N





- Throughput system: ø 10 mm orifice – Surface area P355N chamber = 6333 cm^2
- Background pressure removed
- Pressure drop after 24-hour venting
- Hydrogen outgassing after 12 repeated heating steps 80C/48h:

  7 10^{-16} mbar l s^{-1} cm^{-2}

Silicon coating

aSi:H coating by CVD

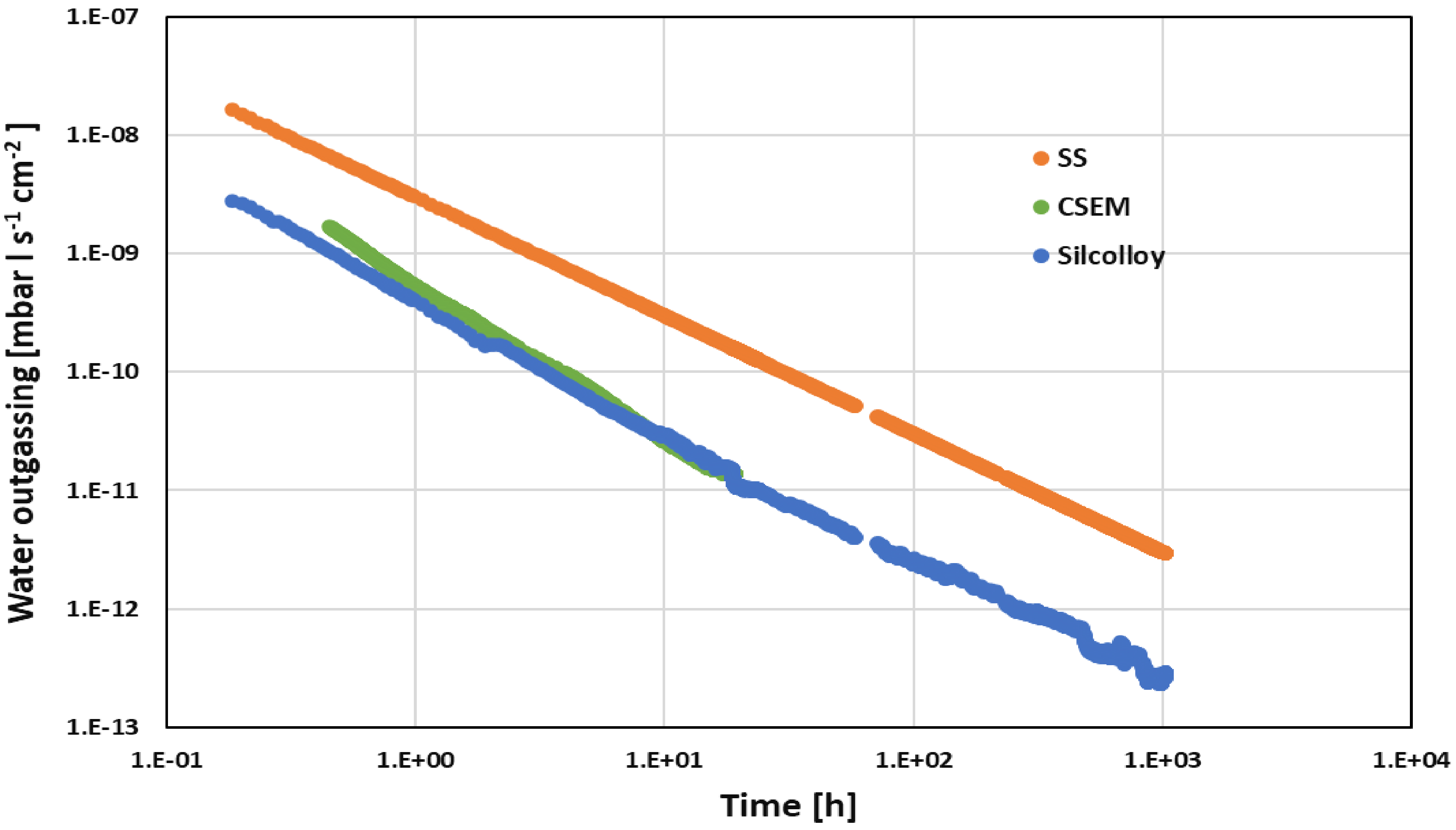
- Thermal enhanced CVD (Silcolloy by SilcoTek)
- Temperature during coating ≈ 400^{o}C
- Coating thickness ≈ 890 nm
- Substrate: 304L – 2 mm – VF* components
  304L – 1 mm – VF* plates

- Plasma enhanced CVD (CSEM)
- Temperature during coating ≈ 180^{o}C
- Coating thickness ≈ 200 nm **(to be confirmed)**
- Substrate: 304L – 0.5 mm – VF* plates



*VF: Vacuum fired

Silcolloy vs CSEM: Pumpdown



Silcolloy:

Q_{10h} = 2.5 10^{-11} mbarl s^{-1} cm^{-2}

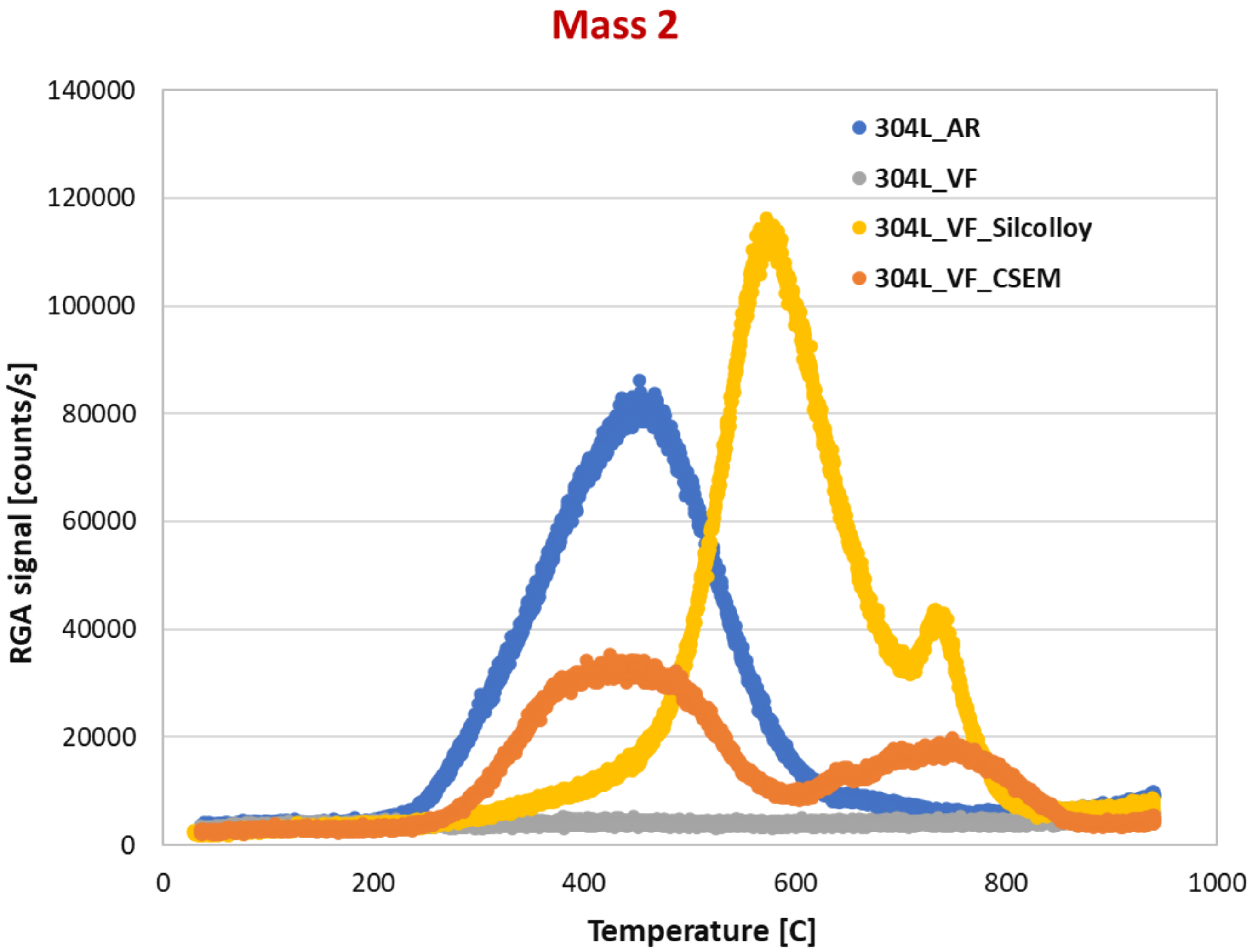
CSEM:

Q_{10h} = 2.6 10^{-11} mbarl s^{-1} cm^{-2}

SS:

Q_{10h} = 3 10^{-10} mbarl s^{-1} cm^{-2}

Silcolloy vs CSEM: TDS



304L_AR — 62.0 ppm (atomic)

⇩

304L_VF — 1.4 ppm (atomic)

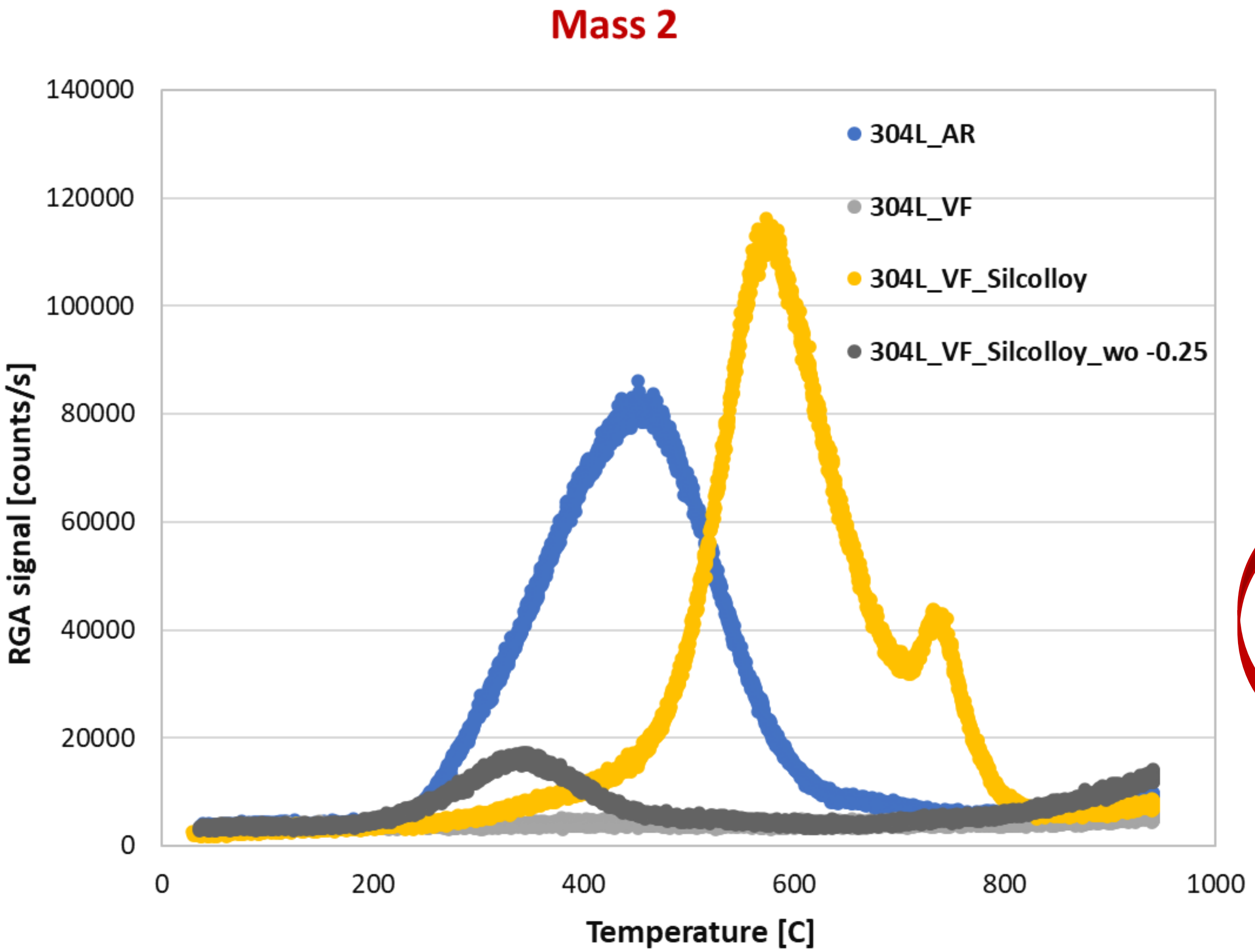
⇩

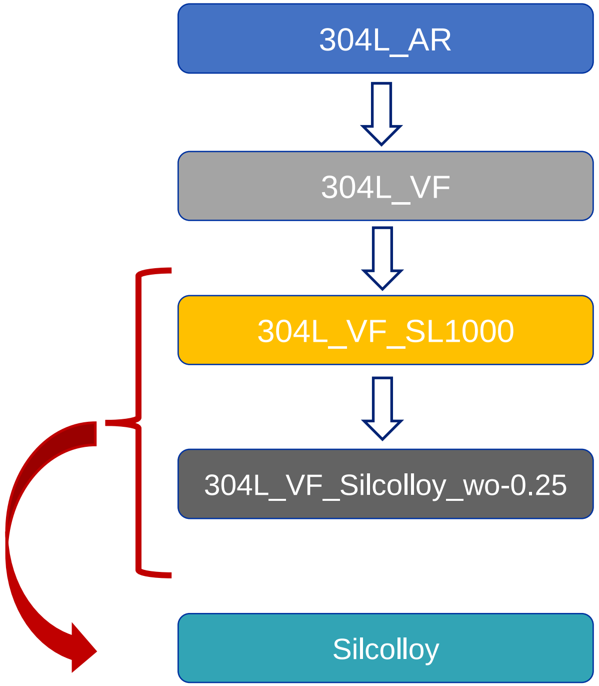
304L_VF_Silcolloy — 81.5 ppm (atomic)

304L_VF_SCEM — 17.9 ppm (atomic)

AR : As Received
VF : Vacuum Fired (950C/2h)
Silcolloy coating — Thermal enhanced CVD
CSEM — Plasma enhanced CVD

Silcolloy: TDS





62.0 ppm (atomic)

1.4 ppm (atomic)

81.5 ppm (atomic)

20.4 ppm (atomic)

≈ 47600 ppm (atomic)

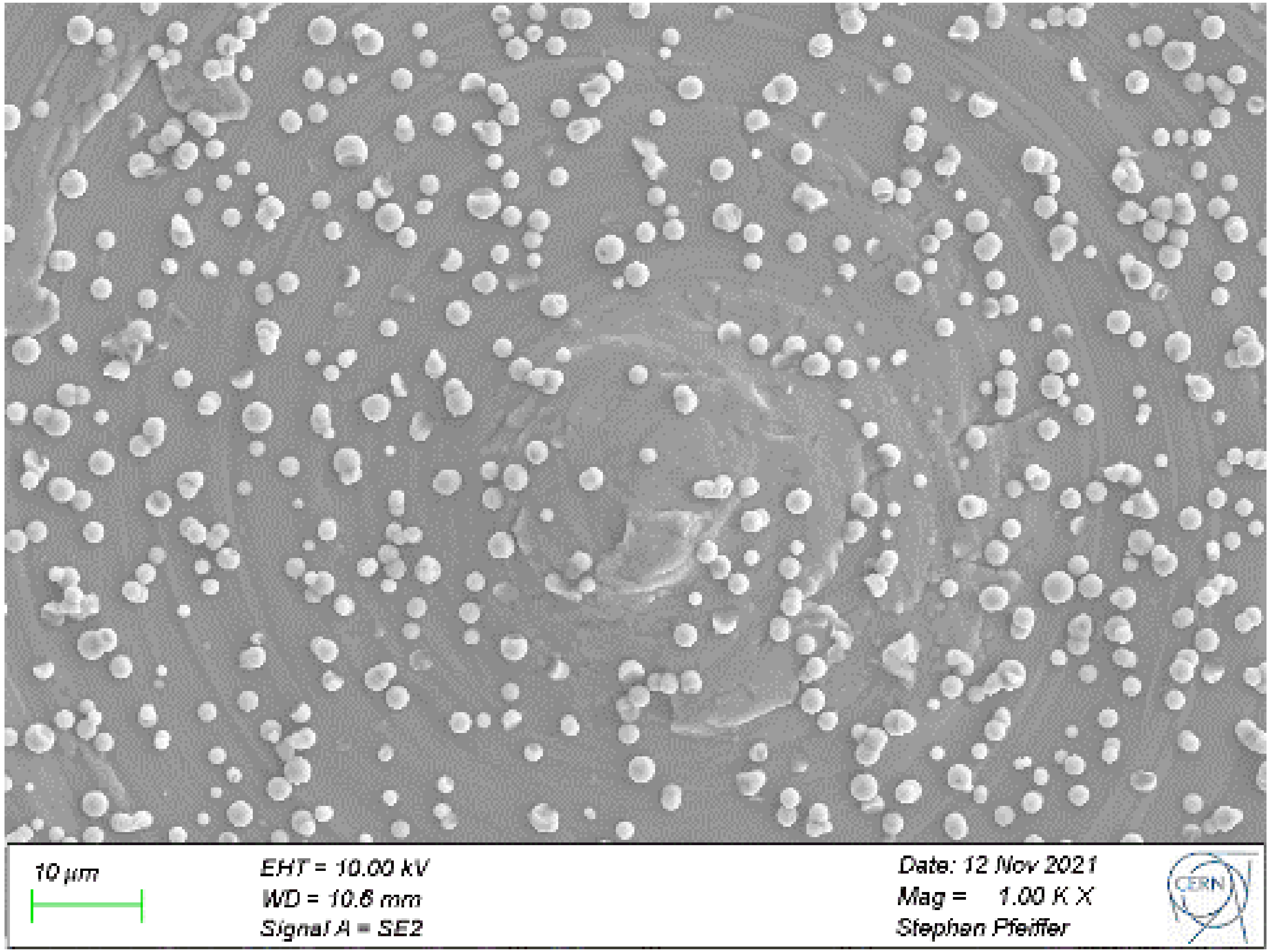
AR : As Received
VF : Vacuum Fired (950C/2h)
Silcolloy coating
wo-0.25 : 0.25 mm removed mechanically

Silcolloy: Hydrogen outgassing

Temperature	Mass	2	15	28	44
80ºC		$7.1\ 10^{-15}$	$3.4\ 10^{-17}$	$1.6\ 10^{-16}$	$2.6\ 10^{-17}$
150ºC		$1.6\ 10^{-14}$	$4.0\ 10^{-17}$	$1.2\ 10^{-16}$	$3.2\ 10^{-17}$
200ºC		$1.5\ 10^{-14}$	$2.0\ 10^{-17}$	$9.9\ 10^{-17}$	$3.9\ 10^{-17}$

- **Outgassing rates units: mbarl s^{-1} cm^{-2}**
- **Duration of heating: 48h**
- **CSEM samples: measurements ongoing**

Silcolloy: SEM



- Spherical particle growth
- Diameter ranging from 1 to 3µm
- Unacceptable for the operation of a gravitational wave detector

Conclusions – mild steel

- The residual water outgassing rate from mild steels were found to be similar or higher (up to 2 orders of magnitude) than that of stainless steel. This large range of water outgassing rates can be attributed to the surface morphology.

- The hydrogen content of mild steel is comparable to those found in vacuum fired stainless steel resulting in low hydrogen outgassing rates.

- Long duration bake-out at 80^{o}C could be used to achieve the pressures required in the next generation of gravitational wave detectors. The effect of such a bake-out on mild steel needs further investigation.

- Simulations of bake-out/pumpdown cycles are now possible. The data has been fitted according to the Temkin isotherm to determine the interval of water binding energies.

Conclusions – aSi:H

- The water outgassing rate is about one order of magnitude lower than standard stainless steel

- The coating contains a huge quantity of hydrogen, and the coating process recharges the stainless-steel substrate

- The outgassing rate for hydrogen is in the lower 10^{-14} mbar l s^{-1} cm^{-2} range. An anomalous increase is measured when increasing the bake-out temperature from $80^{o}C$ to $150^{o}C$

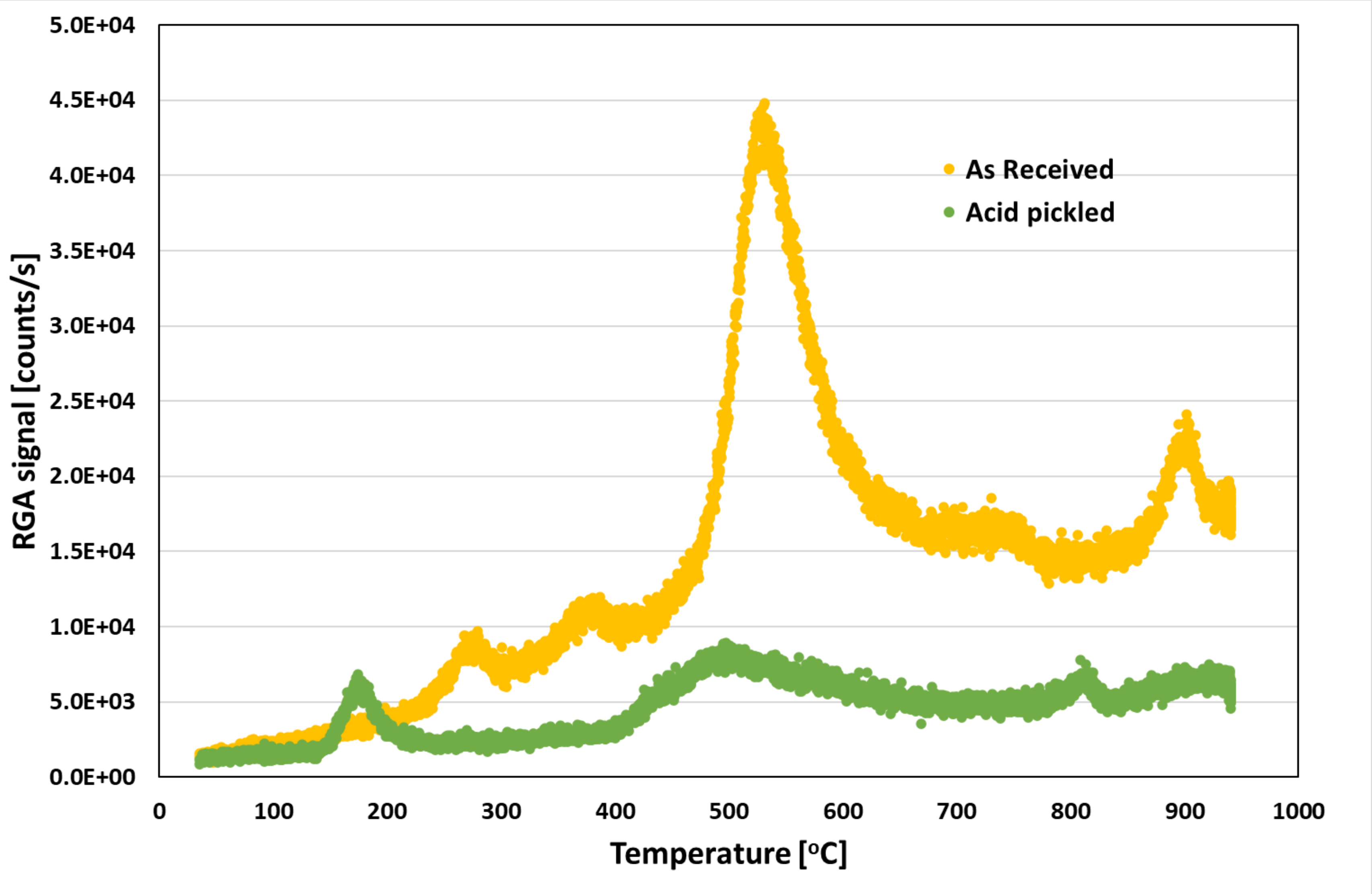
- Spherical particle growth on the surface should be avoided during the coating process

Conclusions

- From a point of view of achievable pressures mild steel is a valid candidate for the manufacturing of the GWD beampipe at a lower cost than the current baseline
- Other important aspects of mild steel must be investigated: long term corrosion resistance, weldability, cleaning methods,…
- The low 80ºC bake-out of mild steel would reduce the cost of materials, energy and insulation
- In the present state of development, despite a reduction of a factor 10 in water outgassing rate, the tested hydrogenated amorphous silicon coating will not avoid the need for a bake-out of the beampipe of future GWD.
- The upsizing this coating technology to future GWD is a technological challenge.

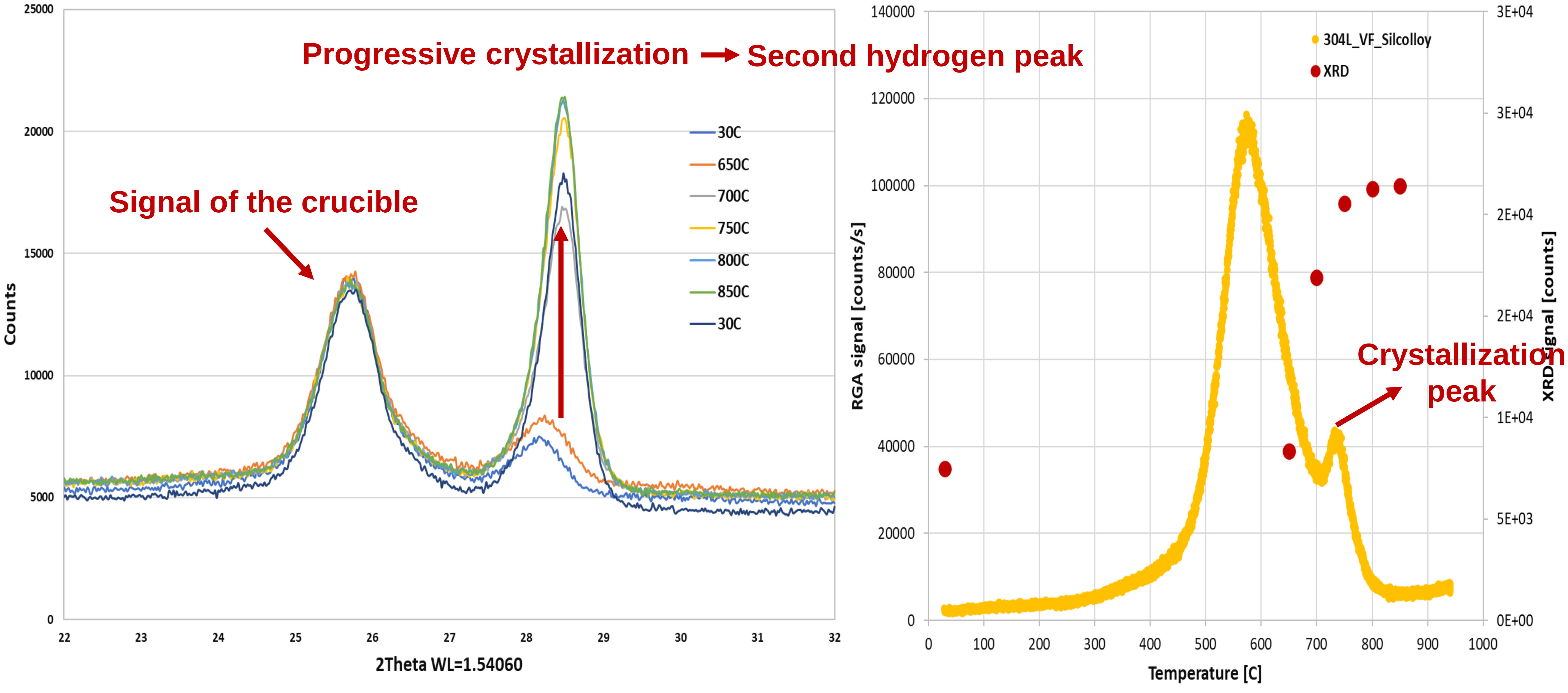
Thank you for your attention

S355J2H: Thermal desorption measurement



Removal of oxide layer by
HCl acid pickling

Silcolloy: XRD



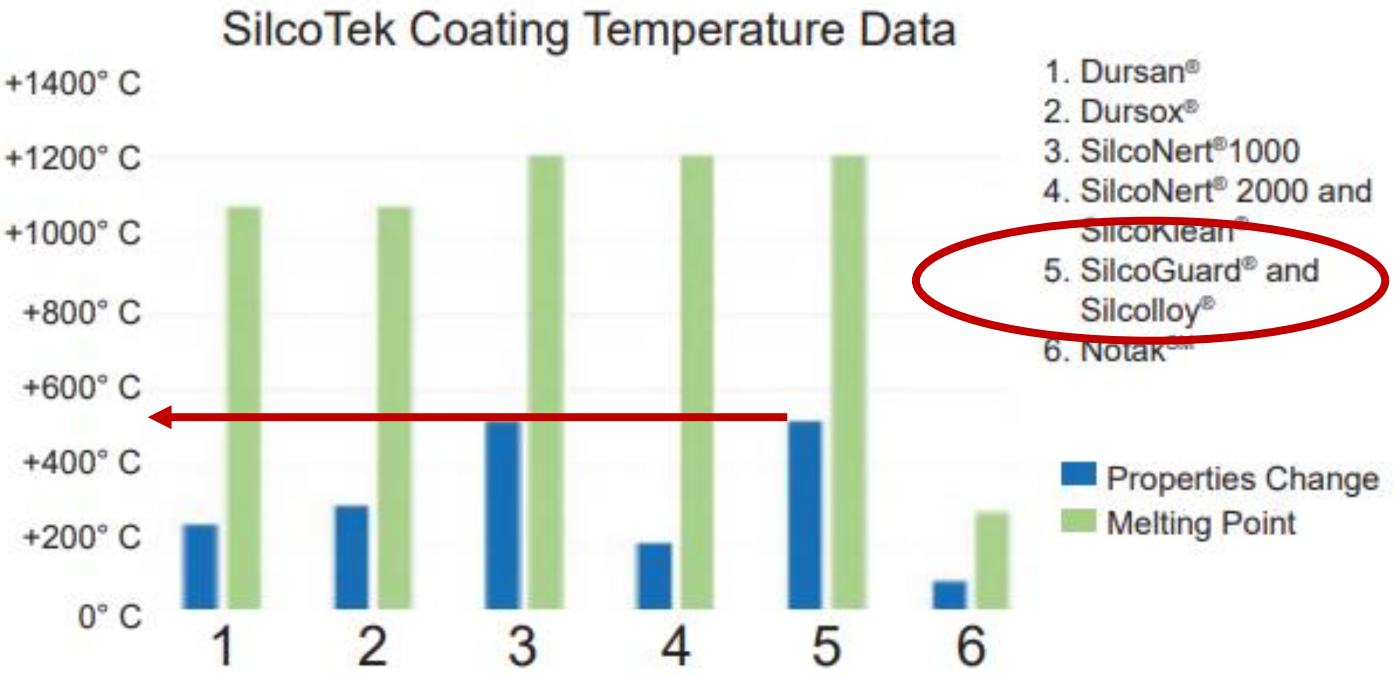
Silcolloy: aSi:H coating by CVD

Silcolloy® Properties

Coating Structure:	Hydrogenated amor
Deposition Process:	Thermal chemical va
Maximum Temperature:*	Advertised propertie Melting: 1410° C
Substrate:	Compatibility: Size: Typical Geometry:
Typical Thickness:	180 - 800 nm
Hydrophobicity (contact angle):	≥40°
Allowable pH Exposure:	0 - 8

HIGH-TEMPERATURE STABLE

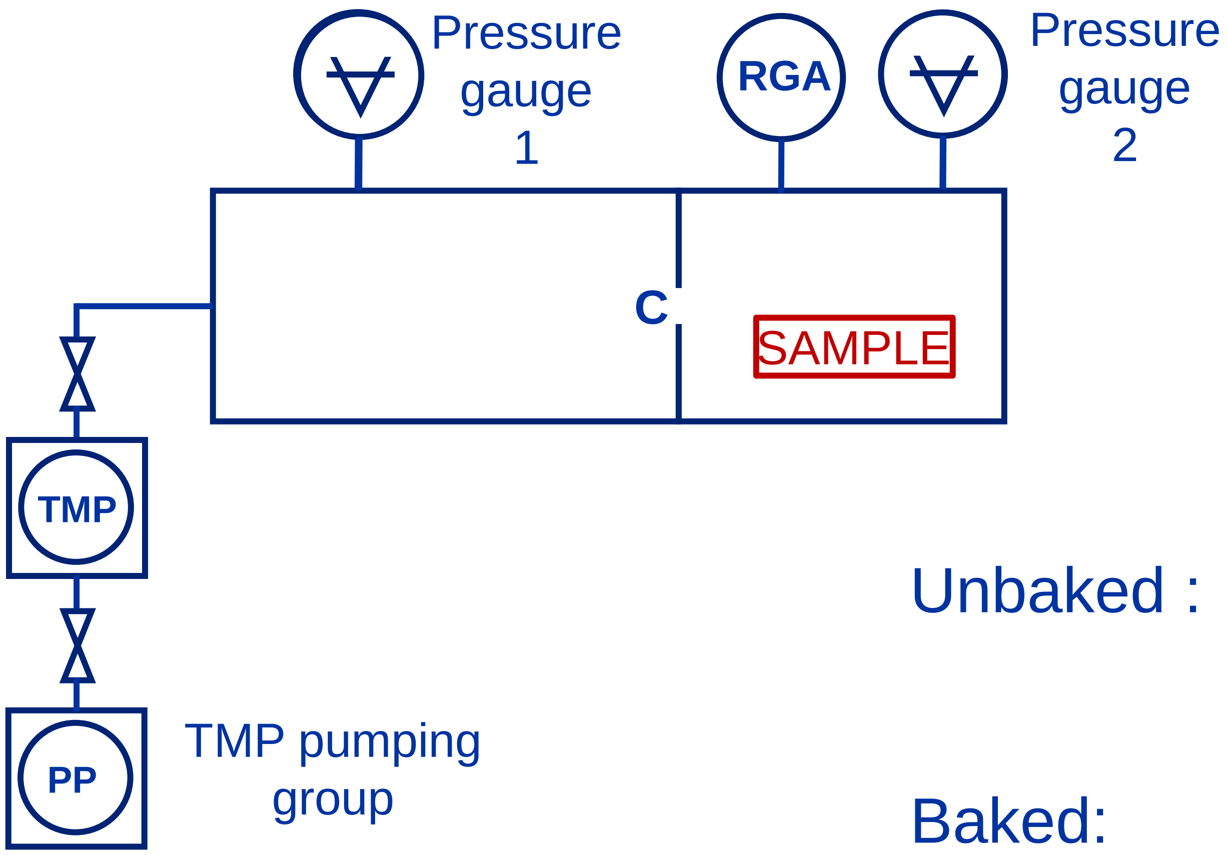
The Silcolloy process provides coatings inert at temperatures up to 1410°C, allowing high temperature analytical or general barrier applications.



* Datasheet from www.silcoTek.com

Material characterization methods

Measurement methods: Throughput



Sample & background — background

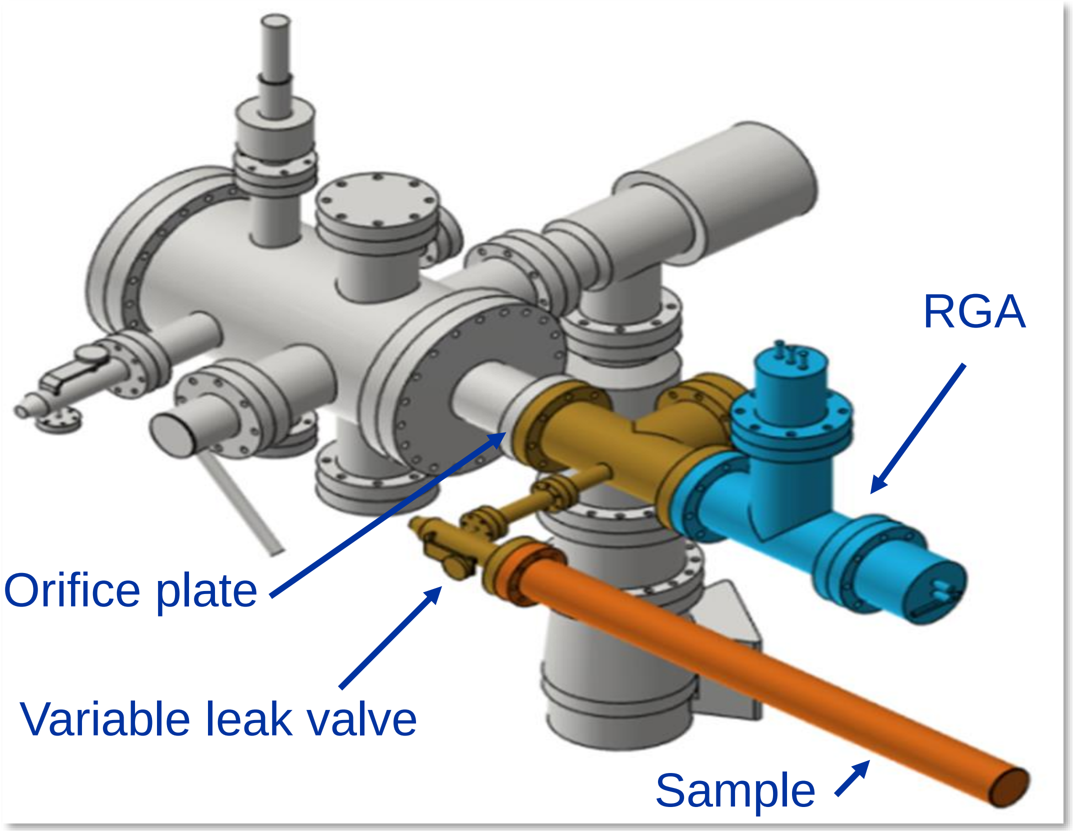
$$Q = \left(\frac{C\,(P_2 - P_1) - C\,(P_{2bg} - P_{1bg})}{A_{sample}} \right)$$

Q in mbar l s^{-1} cm^{-2}

Unbaked : outgassing of water – time dependent
Cold cathode ionization gauges

Baked: outgassing of (mainly) hydrogen
hot cathode ionization gauges

Measurement methods: Coupled-method



All system components are baked to temperatures ranging from 200ºC to 350ºC

Sample and variable leak valve are heated to requested temperature and duration

Prior to cooldown all instrumentation is degassed

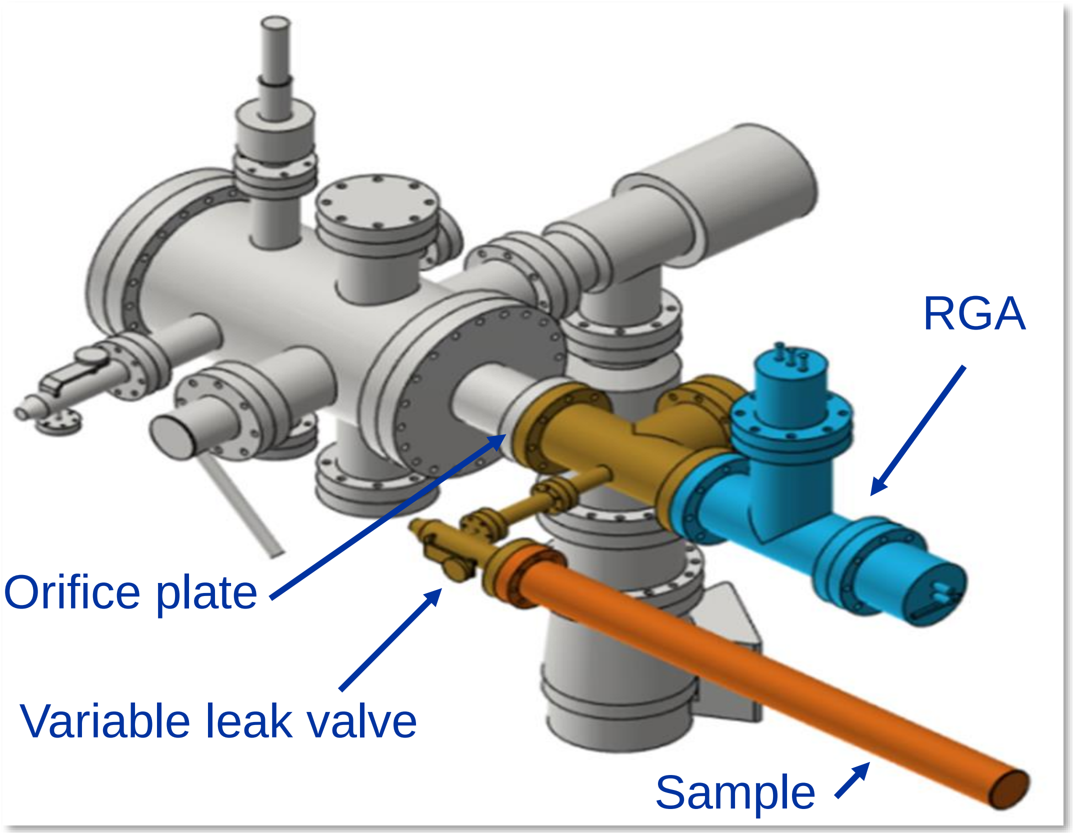
Variable leak valve body has been vacuum fired to minimize contribution

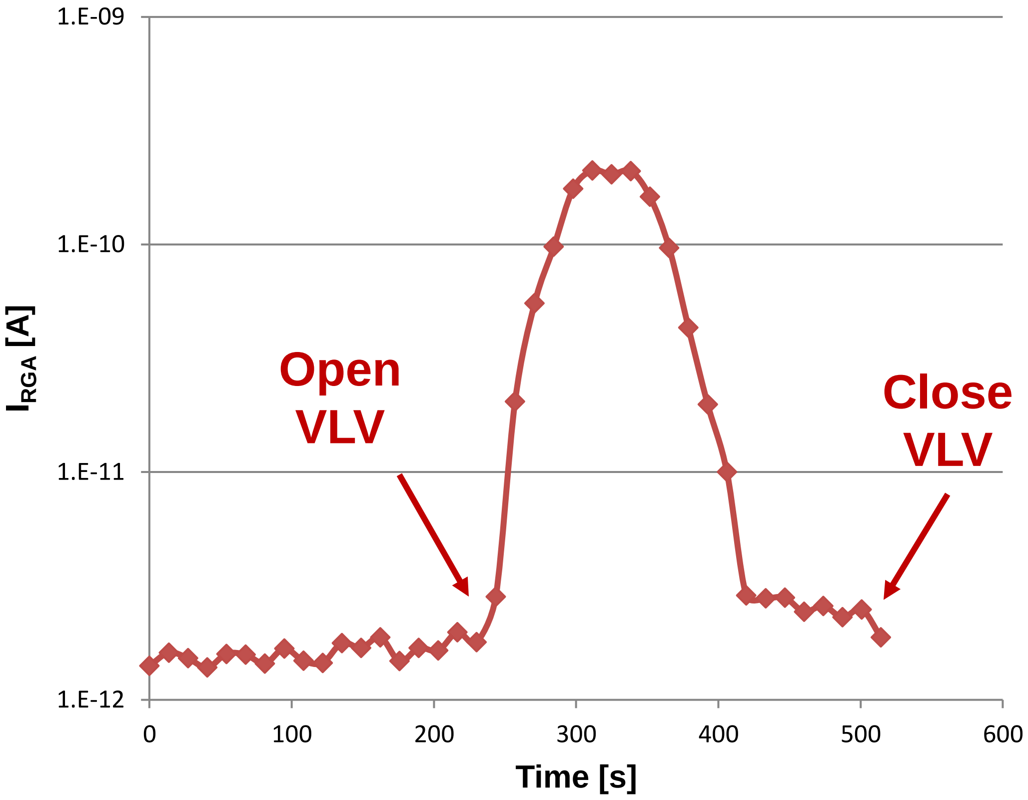
24 hours at room temperature before the start of the first measurement

Not suitable for gasses that can be reabsorbed (water)

RGA has been calibrated to an in-house calibrated hot cathode ionization gauge

Measurement methods: Coupled-method





Measurement methods: Coupled-method

$$Q\ [mbarl/s] = \frac{S_c \times \int_{\Delta\tau} I_{RGA} \times \alpha_{RGA}\, d\tau}{\Delta t_a}$$

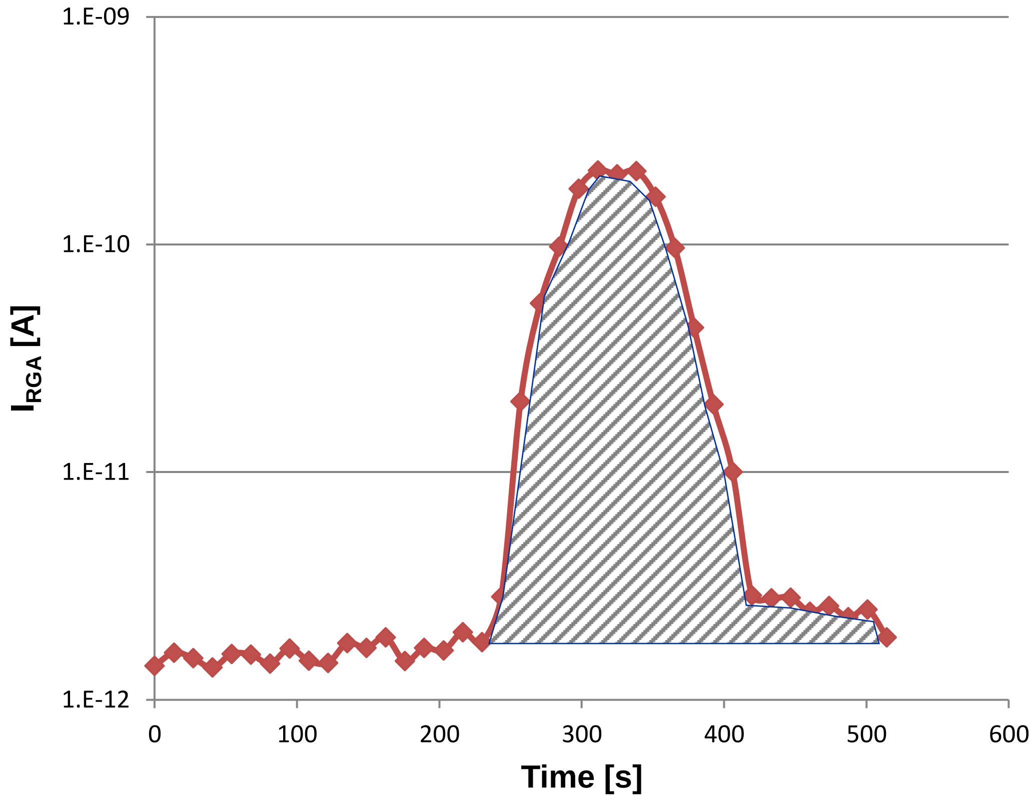
Δt_a: duration of the accumulation [s]

I_{RGA}: current recorded for the gas of interest [A]

S_c: conductance of the orifice for the gas of interest [l/s]

α_{RGA}: calibration factor for the gas of interest [A/mbar]

$\Delta\tau$: duration of the RGA recording [s]



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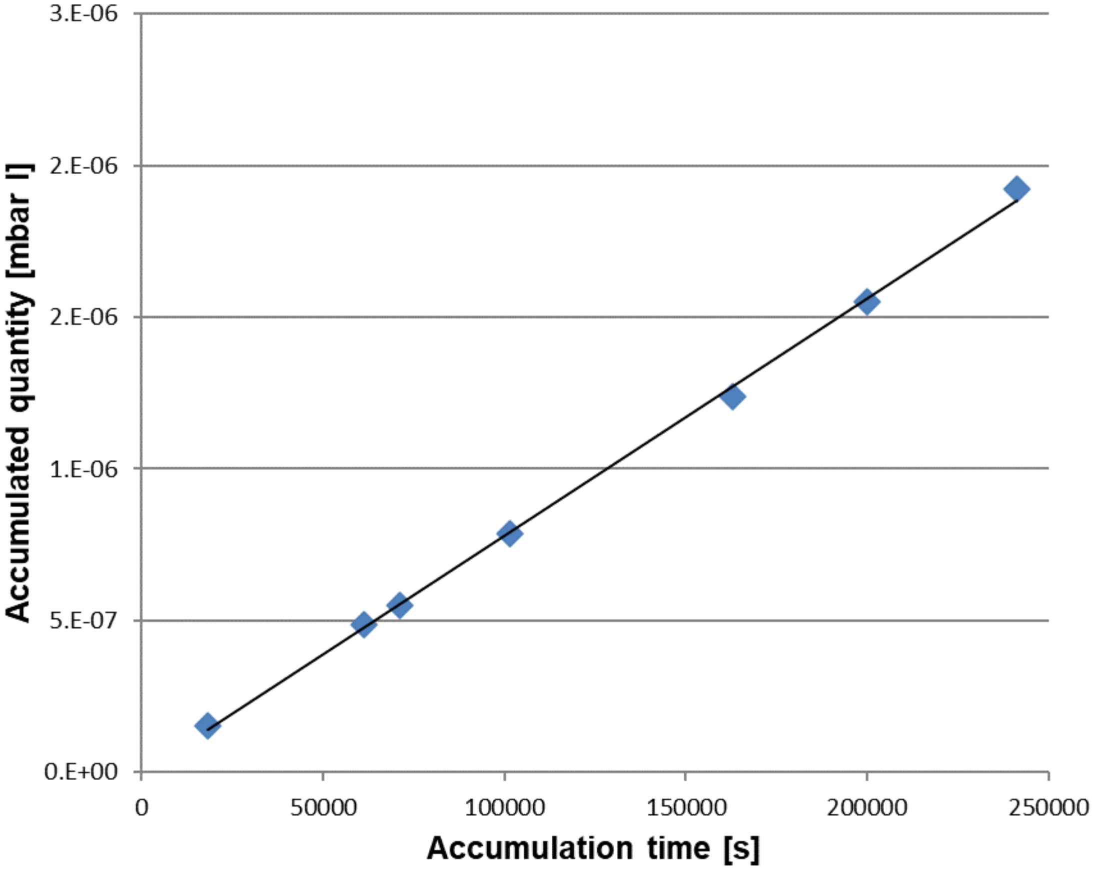
Background outgassing rates (hydrogen)

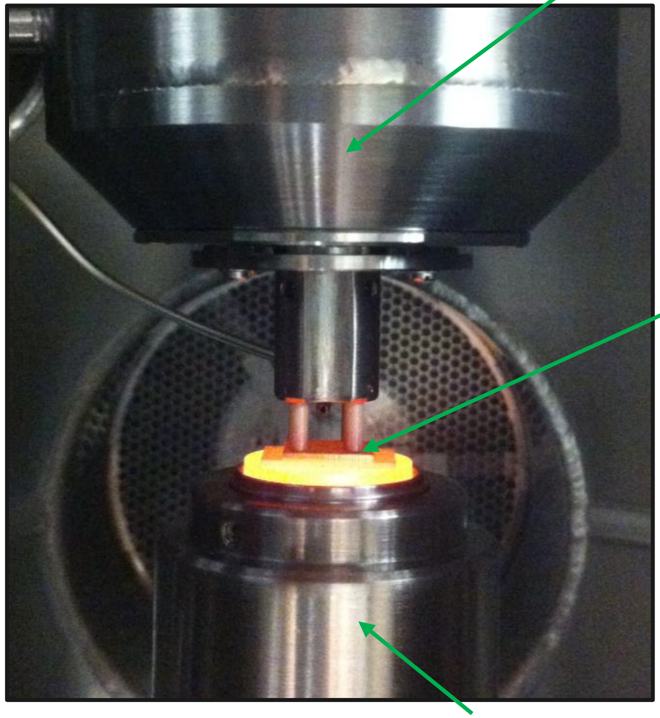
System tubes: 5.3 10^{-13} mbar l s^{-1}

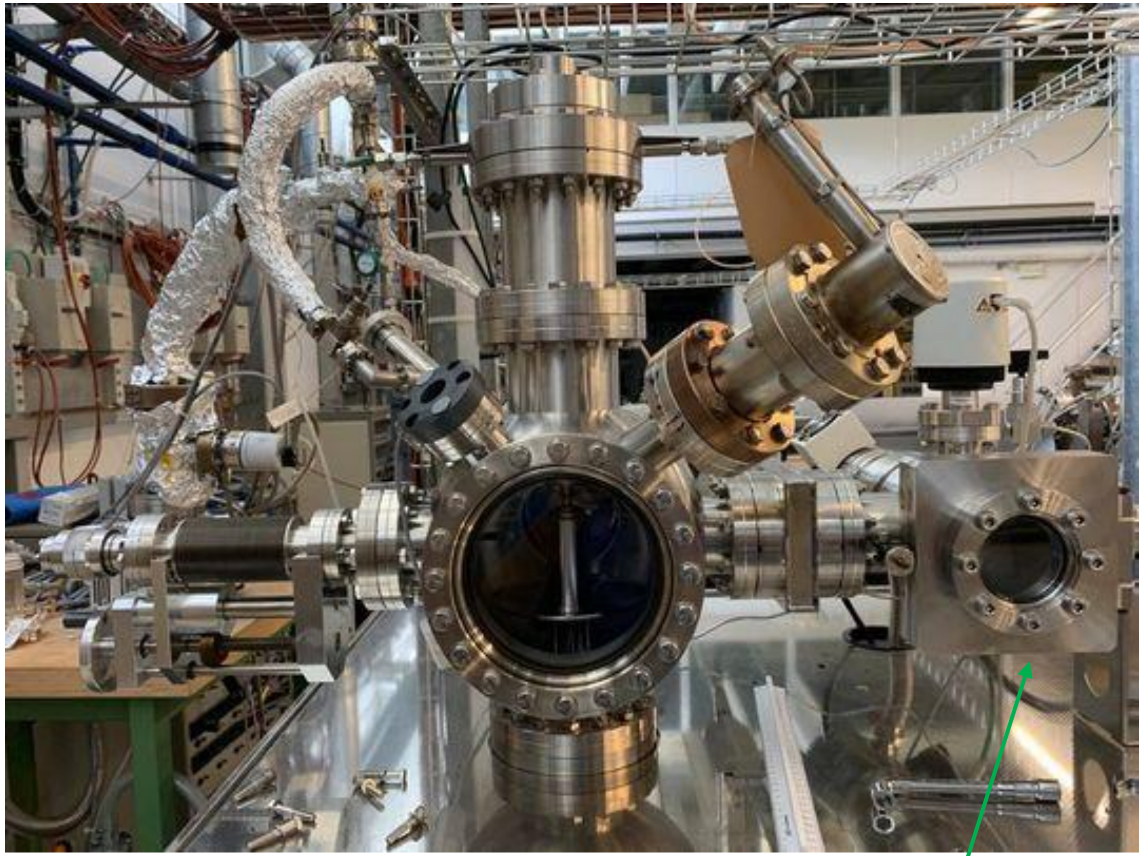
System samples: 7.8 10^{-13} mbar l s^{-1}

Measurement error: ± 40%



Measurement methods: Thermal Desorption





Heating rate: 5K/min

Dimensions of sample: 10mm x 10mm x 1mm

Water binding energy