NEG Coating Studies at DESY: Results and Challenges

Rūta Širvinskaitė, Lutz Lilje, Ralph Böspflug, Sergey Antipov, Nils Plambeck, Marco Schröder, Anna Winiarska
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HELMHOLTZ

DESY.

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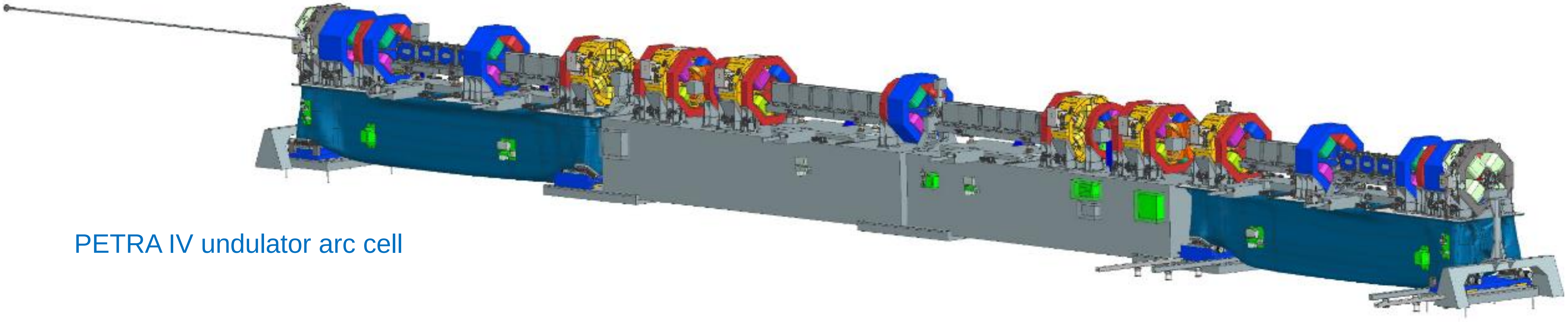
Motivation

Requirements for PETRA IV

- Non-evaporable getter (NEG) coating provides distributed pumping speed as well as desorption barrier, resulting in more uniform pressure profile, reduction of pumps and simpler machine design
- Modern particle accelerators, where most chambers have small diameter and low aspect ratio, rely on NEG increasingly more
- This is the case in PETRA IV, where more than 80 % of the vacuum system will be NEG coated
- PETRA IV aims for a state-of-the-art storage ring that will deliver electron beams of unmatched brightness and quality for photon science experiments



PETRA IV undulator arc cell

NEG studies at DESY

Work towards NEG film vacuum properties optimisation and performance evaluation includes studying:

- Morphology: dense and columnar coating deposition
- Elemental composition: ternary TiZrV and single metal Zr coatings (no alloys targets used)
- Thickness along the chamber
- Sticking probability/capacity measurements
- Microwave signal attenuation measurements

→ Surface preparation as well as good understanding of deposition process/parameters are of high importance!

Some of the more challenging chambers include:

- 5 m long stainless steel undulator dummy chamber
- Copper tapers (sent for tests to MAX IV)

→ Various types of chambers needed in PETRA IV design

- Undulator, arc, extraction chambers most challenging!

PETRA IV vacuum system

High aspect ratio chambers

Undulator chambers

- 4 different types
- Amount: 34
- Length: 4550-4291 mm
- Inner dimension: 20x7 mm ellipse (26x7 canting)
- Material: Aluminum EN AW-6060 (AlMgSi0.5)

Wiggler chambers

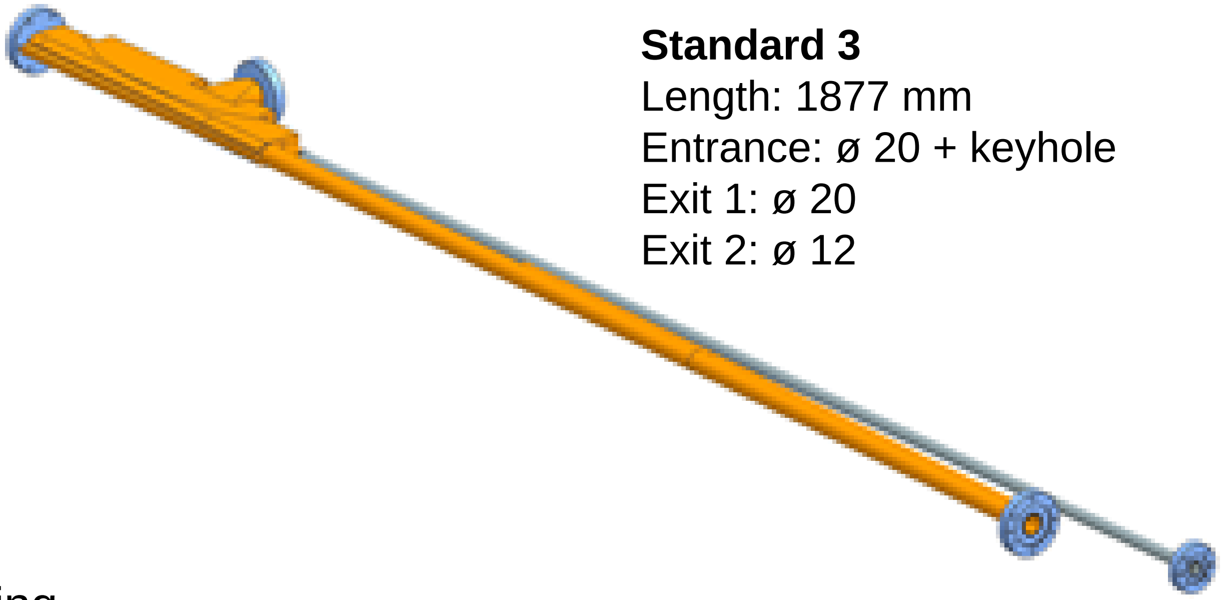
- Amount: 40
- Length: 4291 mm
- Inner dimension: 20x13 mm ellipse
- Material: Copper CuAg0.10(OF) (CW019A)
- Flange area: CuCr1Zr (CW106C)

PETRA IV vacuum system

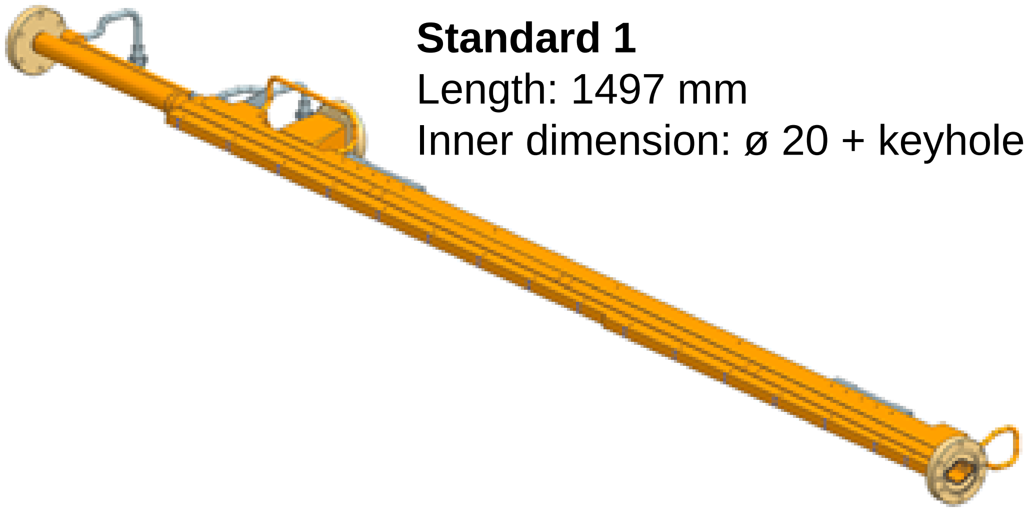
Photon extraction chambers (three standard types)

Photon extraction chamber 1, 2 and 3 standard

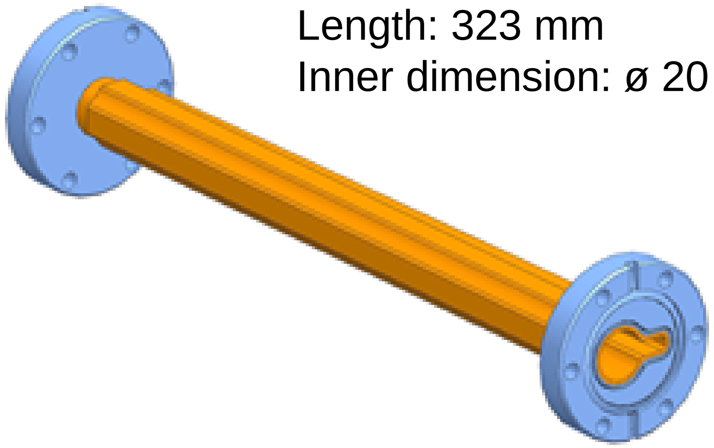
- Amount: 67 each
- Material: Copper CuAg0.10(OF) (CW019A)
- Flange area: CuCr1Zr (CW106C)
- Few more (less occurring) types will need NEG coating



Standard 3
Length: 1877 mm
Entrance: ø 20 + keyhole
Exit 1: ø 20
Exit 2: ø 12



Standard 1
Length: 1497 mm
Inner dimension: ø 20 + keyhole



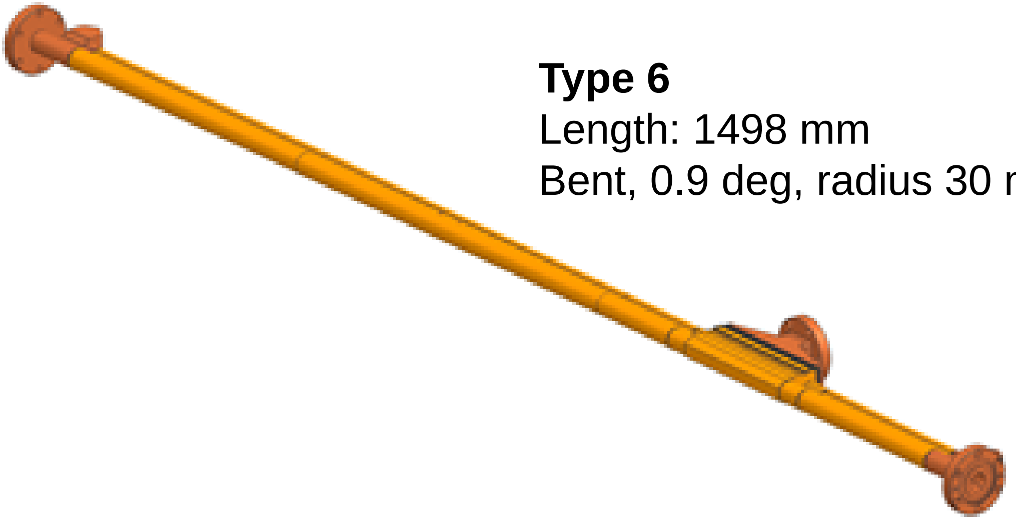
Standard 2
Length: 323 mm
Inner dimension: ø 20 + keyhole

PETRA IV vacuum system

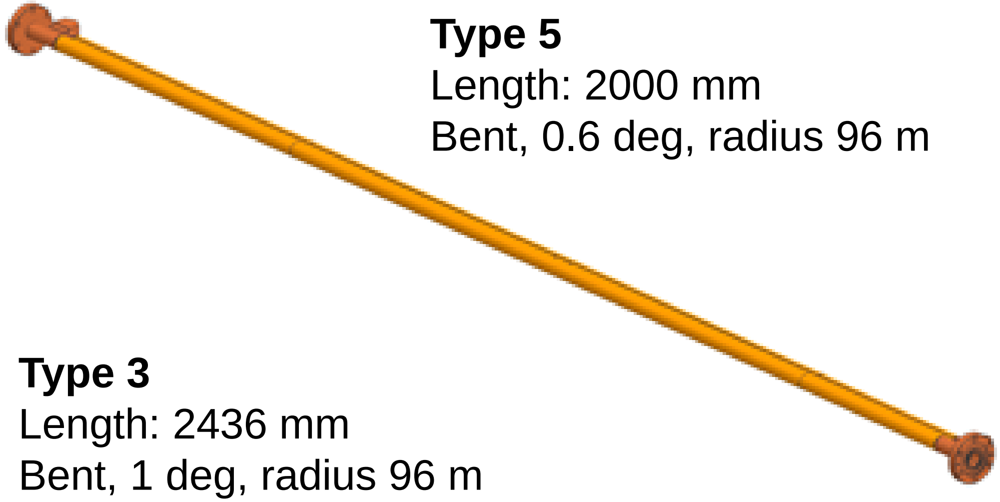
Bent chambers

Arc chambers (6 main types; 4 bent)

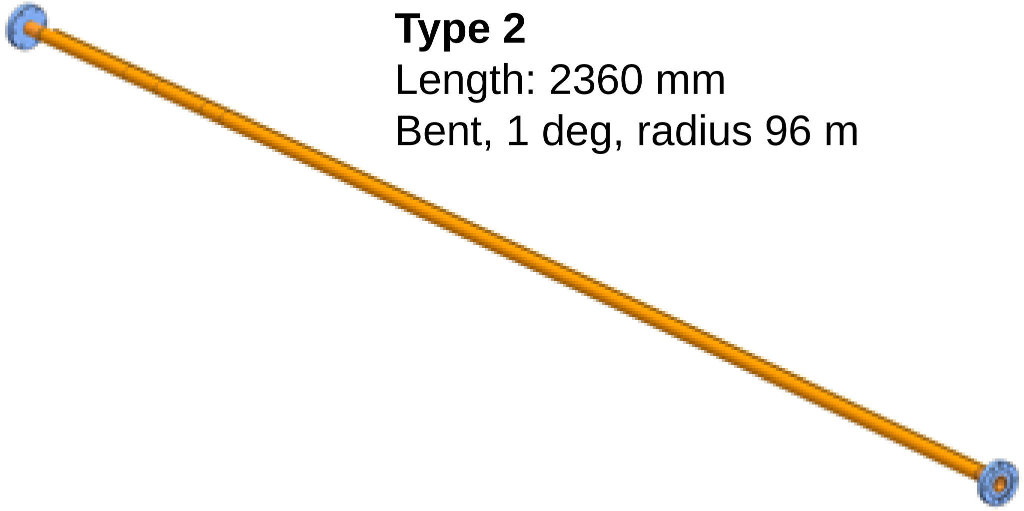
- Amount: 72 each (68 type 4)
- Material: Copper CuAg0.10(OF) (CW019A)
- Flange area: CuCr1Zr (CW106C)
- Inner dimension: ø 20
- Shadowing bumps at the end



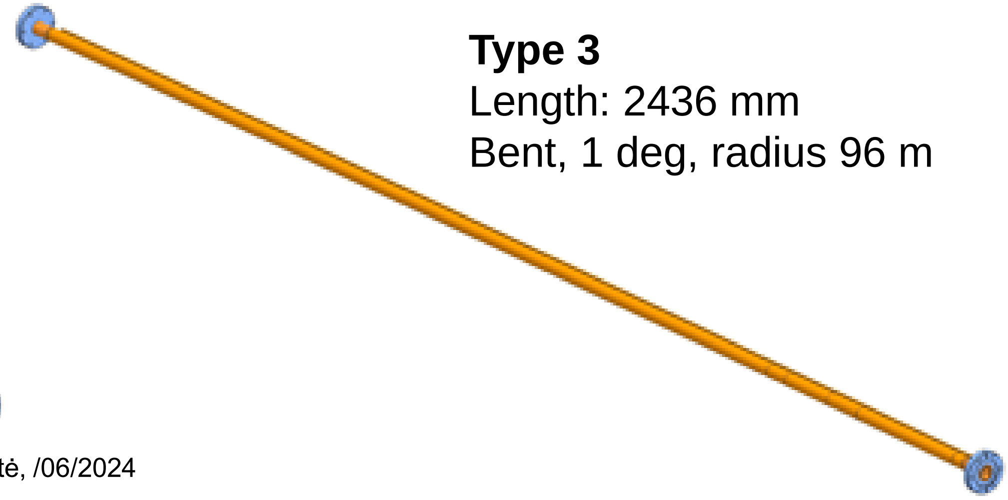
Type 6
Length: 1498 mm
Bent, 0.9 deg, radius 30 m



Type 5
Length: 2000 mm
Bent, 0.6 deg, radius 96 m



Type 2
Length: 2360 mm
Bent, 1 deg, radius 96 m



Type 3
Length: 2436 mm
Bent, 1 deg, radius 96 m

Standard Samples

Definition

- Standard test samples:
  - Material – OFS-Cu (previously OFHC-Cu)
  - Flanges – 316 LN
  - Length – 1 m
  - Dinner diameter – 20 mm

- Sufficient complexity and similarity to PETRA IV chambers
- Welding, cleaning, etching done on site
- Significantly different procedures for both substrates
  - Different gases used for welding
  - Different etching procedures needed

- 50 cm 20 mm ID samples used for resistivity and (eventually) ESD measurements

Surface treatments at DESY

General procedure for OFHC-Cu

Production
- Welding flanges to pipes (Formier gas used; 95% N_2 + 5% H_2)

→ Days/Weeks

Degreasing
- Isopropanol wiping
- ElmaClean (EC) 115c 15 mins in US bath
- Isopropanol rinsing

→ Days/Weeks

Weld pre-treatment
- Etching with sulphuric acid-based solutions (various durations)
- ElmaClean 115c 5-15 mins in US bath

→ 20 hours

Etching
- Ammonium peroxid sulphate etching
- ElmaClean 115c 5 mins
- Isopropanol rinsing

→ 2 hours

Weld post-treatment
- Passivation with chromic acid solution (various durations)
- ElmaClean 115c 5 mins in US bath

→ 2-20 hours

Coating facility
- Installation
- Bakeout
- Deposition

→ Days/weeks

Vacuum tests
- Sticking probability
- Capacity

→ Days/weeks

Non-vacuum tests
- Resistivity
- Thickness
- Morphology
- Elemental composition

Clean/coated samples stored in N_2 cabinet

Sample preparation

Degreasing and etching

- Chamber material – **OFHC-Cu** (CW022A)
- The following cleaning procedure was applied to the samples:

Degreasing

- ElmaClean (1.5%) in US bath for 15 min at 65 °C
- Demineralized water (DMW) rinsing (until the conductivity drops to <0.1 µS/m) (around 4 min)
- Isopropanol rinsing (pouring into the tube)
- N_2 drying (5 min)
- Air dryer (60 °C for 2 hours)

Etching

- DMW rinsing - 2 min (or until the conductivity drops down below 0.4 µS/cm)
- Ammonium persulphate $(NH_4)_2S_2O_8$ (250g/l) - 45 min at room temperature
- DMW rinsing → N_2 drying → DMW rinsing (each two min)
- Elma Clean 1.5% - 5 min at 65 ºC
- DMW rinsing → N_2 drying → DMW rinsing (each two min)
- N_2 drying - 5 mins
- Isopropanol rinsing - 1 min
- N_2 drying - 10 mins

Surface treatments at DESY

General procedure for OFS-Cu

Production
- Welding flanges to pipes (Ar gas used)

Degreasing
- Isopropanol wiping
- ElmaClean (EC) 115c 15 mins in US bath
- Isopropanol rinsing

Etching
- Ammonium peroxid sulphate + ammonium citrate etching
- Hydrogen peroxide
- Ammonium acetate

Coating facility
- Installation
- Bakeout
- Deposition

Vacuum tests
- Sticking probability
- Capacity
- Ageing
- *ESD*

Non-vacuum tests
- Resistivity
- Thickness
- Morphology
- Elemental composition

Clean/coated samples stored in N_2 cabinet

Sample preparation

Degreasing and etching

- Chamber material – **OFS-Cu** CW019A (CuAg0.10(OF))
- The following cleaning procedure was applied to the samples:

Degreasing

- ElmaClean (1.5%) in US bath for 15 min at 65 °C
- Demineralized water (DMW) rinsing (until the conductivity drops to <0.1 µS/m) (around 4 min)
- Isopropanol rinsing (pouring into the tube)
- N_2 drying (5 min)
- Air dryer (60 °C for 2 hours)

Etching

- DMW rinsing - 2 min (or until the conductivity drops down below 0.4 µS/cm)
- Ammonium persulphate $(NH_4)_2S_2O_8$ (100g/l) + ammonium citrate $NH_4CH_3CO_2$ (1g/l) - 5 min at room temperature
- DMW rinsing → N_2 drying → DMW rinsing (each two min)
- Hydrogen peroxide H_2O_2 (100g/l) - 10 min at room temperature
- DMW rinsing → N_2 drying → DMW rinsing (each two min)
- Ammonium acetate $C_6H_{17}N_3O_7$ (50 g/l) - 5 min at room temperature
- DMW rinsing → N_2 drying → DMW rinsing (each two min)
- N_2 drying (5 min) → Isopropanol rinsing (1 min) → N_2 drying (10 mins)

OFS-Cu coating

Dense samples

- All dense test samples:

Sample	Target	Etching	Deposition mode	Coating	Thickness (µm)	Set parameter	t (h)	p (mbar)	$I_{solenoid}$ (A)
TK Ag 11	TiZrV	N	Pulsed DC (AEPP)	Dense	1	40 W	5	7e-2	70
TK Ag 12	TiZrV	N	Pulsed DC (AEPP)	Dense	2	50 W	10	7e-2	70
TK Ag 13	TiZrV	Y	Pulsed DC (AEPP)	Dense	1	70 W	5	7e-2	70
TK Ag 15	TiZrV	Y	Pulsed DC (AEPP)	Dense	2	50 W	10	7e-2	70
TK Ag 18	Zr	Y - defect	Pulsed DC (AEPP)	Dense	2	50 W	10	7e-2	70
TK Ag 20	Zr	Y	Pulsed DC (AEPP)	Dense	1	70 W	5	7e-2	70

- Some samples will be used for pumping tests
- Only recently started doing pulsed DC deposition
- 350 kHz, 1.1 µs pulses
- Coating coverage and uniformity OK, process stable at higher power

OFS-Cu coating

Columnar samples

- All columnar test samples:

Sample	Target	Etching	Deposition mode	Coating	Thickness (µm)	Set parameter	t (h)	p (mbar)	$I_{solenoid}$ (A)
TK Ag 06	TiZrV	Y	DC	Columnar	1	200 mA	5.5	5e-1	70
TK Ag 05	TiZrV	Y	DC	Columnar	1	200 mA	5.5	5e-1	72.3
TK Ag 07	TiZrV	Y	DC	Columnar	2	250 mA	10	5e-1	70
TK Ag 08	Zr	Y	DC	Columnar	2	200 mA	10	5e-1	70
TK Ag 09	TiZrV	Y	DC	Columnar	2	250 mA	10.5	2e-1	70
TK Ag 10	TiZrV	Y	DC (AEPP)	Columnar	1	200 mA	5	5e-1	70
TK Ag 14	TiZrV	Y	DC (AEPP)	Columnar	1	70 W	5	5e-1	70
TK Ag 16	TiZrV	Y	DC (AEPP)	Columnar	1	60 W	5	5e-1	70
TK Ag 17	TiZrV	Y	DC (AEPP)	Columnar	2	200 mA	10	2e-1	70
TK Ag 19	Zr	Y	DC (AEPP)	Columnar	2	250 mA	10	5e-1	70
TK Ag 21	Zr	Y	DC (AEPP)	Columnar	1	50 W	5	5e-1	70
TK Ag 22	Zr	Y	DC (AEPP)	Columnar	1	50 W	7	5e-1	70
TK Ag 23	Zr	Y	DC (AEPP)	Columnar	1	55 W	5	5e-1	70
TK Ag 30	TiZrV	Y	DC (AEPP)	Columnar	1	60 W	5	7e-2	70
TK Ag 31	Zr	Y	DC (AEPP)	Columnar	1	60 W	5	7e-2	70

- Some samples will be used for pumping tests

OFS-Cu coating

Challenges when producing columnar coating

1. **Non-uniform thickness**
   - Thickness at least twice as large at the center of the tube when compared to the ends
2. **Uncoated ends**
   - Not uncommon issue with higher aspect ratio tubes
   - Likely due to plasma instability which is affected by the magnetic field/higher pressure
   - Extensive B measurements done, more fans installed to try and increase B (currently up to 500 Gauss in the middle)
   - Temporary fix – moving the magnet down
3. **Unstable process**
   - Difficult to maintain deposition for 5 hours with 70 W at 4.8×10^{-1} mbar

- More sample sets have been produced:
  - Samples for pumping property/resistivity measurements
  - Samples for NEG ageing tests

Assessing pumping properties

Deposition

PVD system and deposition parameters

- 1 m long solenoid magnet (4 × 100 turns) – standard samples are of the same length; however, to enable the sample installation on the microwave test stand for attenuation measurements, 50 cm long tubes (ID 20 mm) were coated
- 3×1 mm wires twisted on site (single metal Ti, Zr and V and single metal Zr)
- Comparing:
  - Substrate (OFHC-Cu vs OFS-Cu)
  - Material: TiZrV vs Zr
  - Thickness: 5 µm vs1 µm
  - Structure: dense vs columnar (not for OFHC-Cu)
- 12 reference samples (two sets) in total prepared

Deposition

PVD system and deposition parameters

- OFHC-Cu samples:

Sample	Mode	Expected thickness	P_{Kr} (mbar)	$I_{solenoid}$ (A)	I (mA)	Duration (h)	Cathode	Estimated thickness
TK 50-09	DC	5	7.2×10^{-2}	70	200	25	TiZrV twisted	6.8
TK 50-10	DC	1	7.2×10^{-2}	70	200	5.5	TiZrV twisted	1.3
TK 50-11	DC	5	7.2×10^{-2}	70	200	25	Zr twisted	5.9
TK 50-12	DC	1	7.2×10^{-2}	70	200	5	Zr twisted	1.4

- OFS-Cu samples:

Sample	Mode	Expected thickness	P_{Kr} (mbar)	$I_{solenoid}$ (A)	P (W)	Duration (h)	Cathode	Estimated thickness
TK Ag 50-09	Pulsed DC	1	7.2×10^{-2}	70	70	5	Zr twisted	1 µm
TK Ag 50-11	DC	1	4.8×10^{-1}	70	50	5	Zr twisted	1 µm
TK Ag 50-10	Pulsed DC	1	7.2×10^{-2}	70	70	5	TiZrV twisted	1 µm
TK Ag 50-14	DC	1	4.8×10^{-1}	70	50	5	TiZrV twisted	1 µm
TK Ag 50-16	Pulsed DC	5	7.2×10^{-2}	70	70	25	Zr twisted	5 µm
TK Ag 50-17	DC	5	4.8×10^{-1}	70	50	11	Zr twisted	5 µm
TK Ag 50-18	Pulsed DC	5	7.2×10^{-2}	70	70	25	TiZrV twisted	5 µm
TK Ag 50-19	DC	5	4.8×10^{-1}	70	50	18	TiZrV twisted	5 µm

Vacuum tests

Setup and procedure

- System allows for pressure ratio measurements (ESD tests are planned in the future)
- Main components can be split into three categories: pumping line, injection line and test domes
- During bakeout, samples were kept at 80 °C with the rest of the system at 200 °C
- System kept at 150 °C for two hours at the start of sample activation (filaments degassed before increasing the sample temperature)
- First activation to 140 °C; subsequent activations to 150, 160, 180, 200, 220 and 250 °C (all 24 hours long)
- Short H_2 injection (5 min) to obtain pressure ratio P_{bot}/P_{top} after each activation
- Followed by a longer CO injection (until pressure ratio drops to < 10) to saturate the film

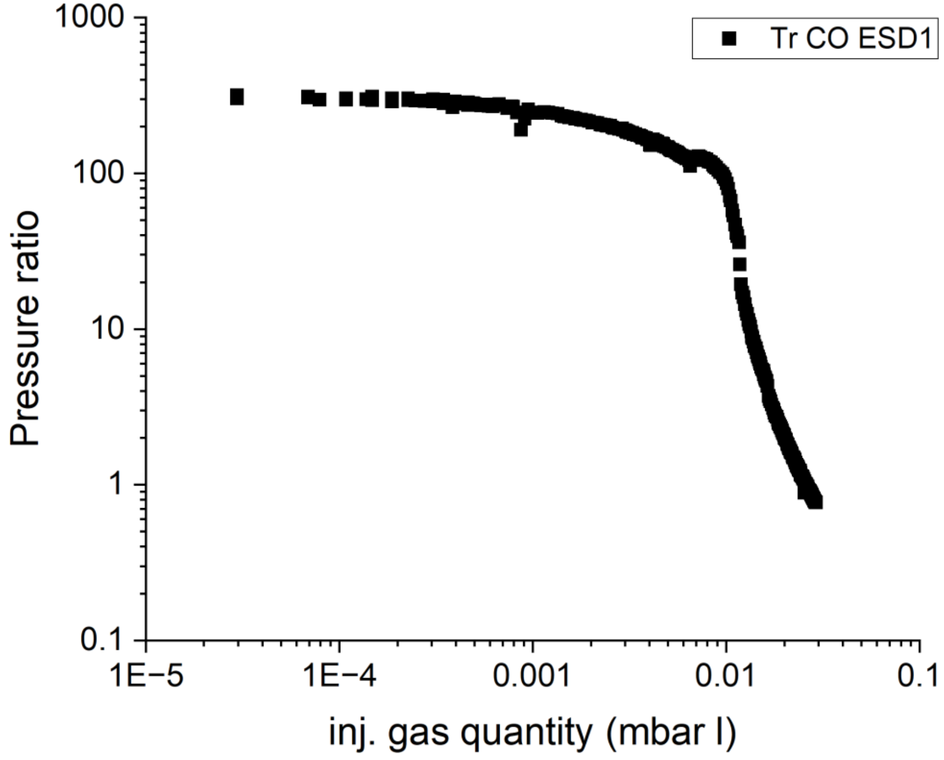
Bakeout/activation procedure

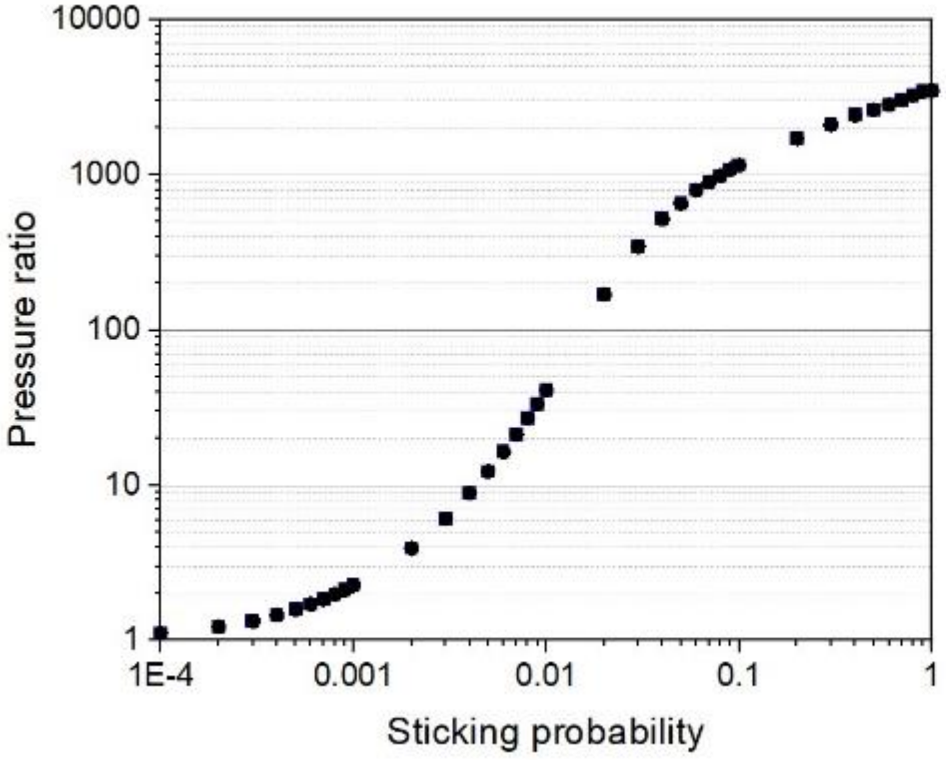
Injection procedure

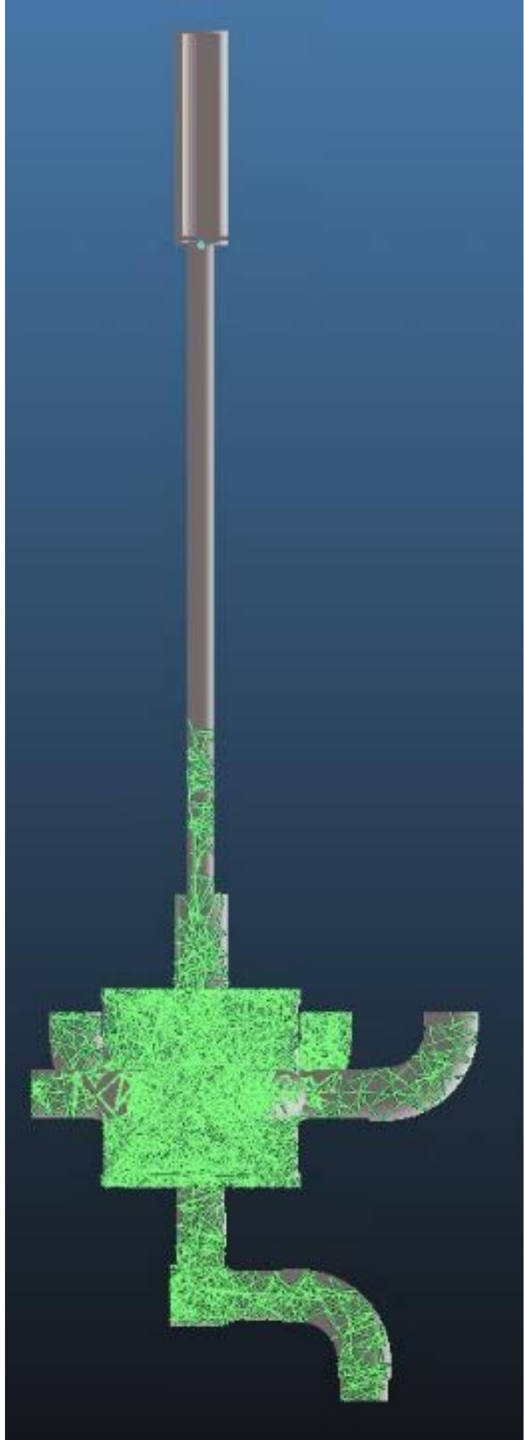
Vacuum tests

Sticking probability and pumping capacity

- Only 1 µm samples were used for measurements
- With the use of Molflow, P_{bot}/P_{top} can be converted to sticking probability
- The amount of injected CO before P_{bot}/P_{top} decreases below 10 represents CO pumping capacity





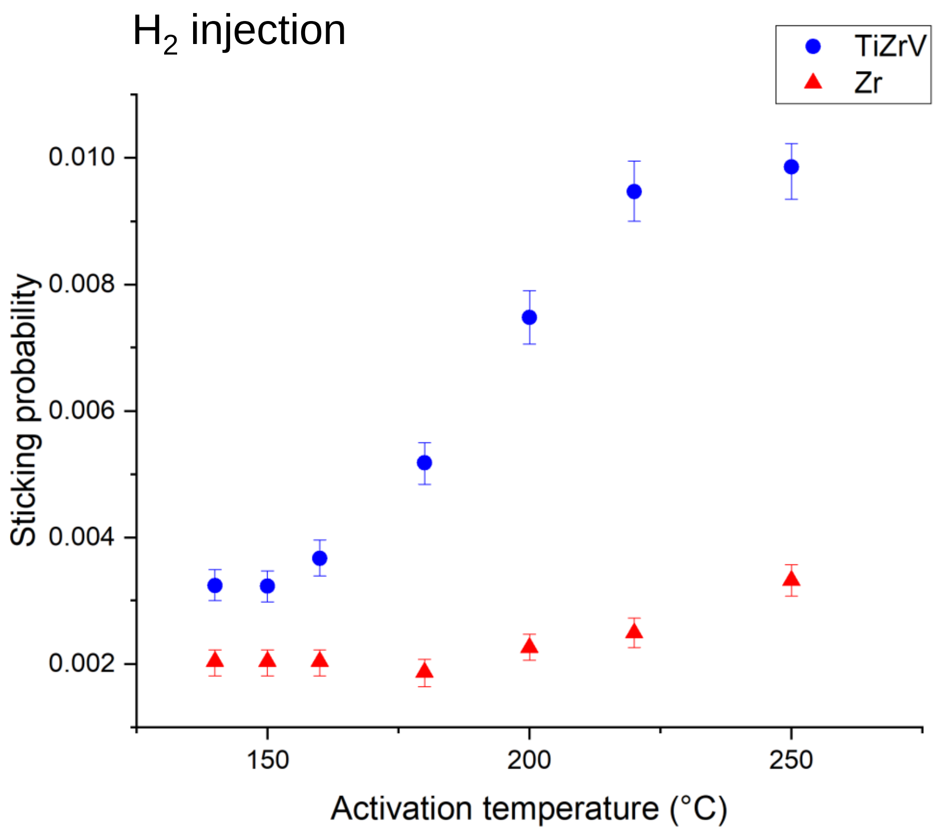


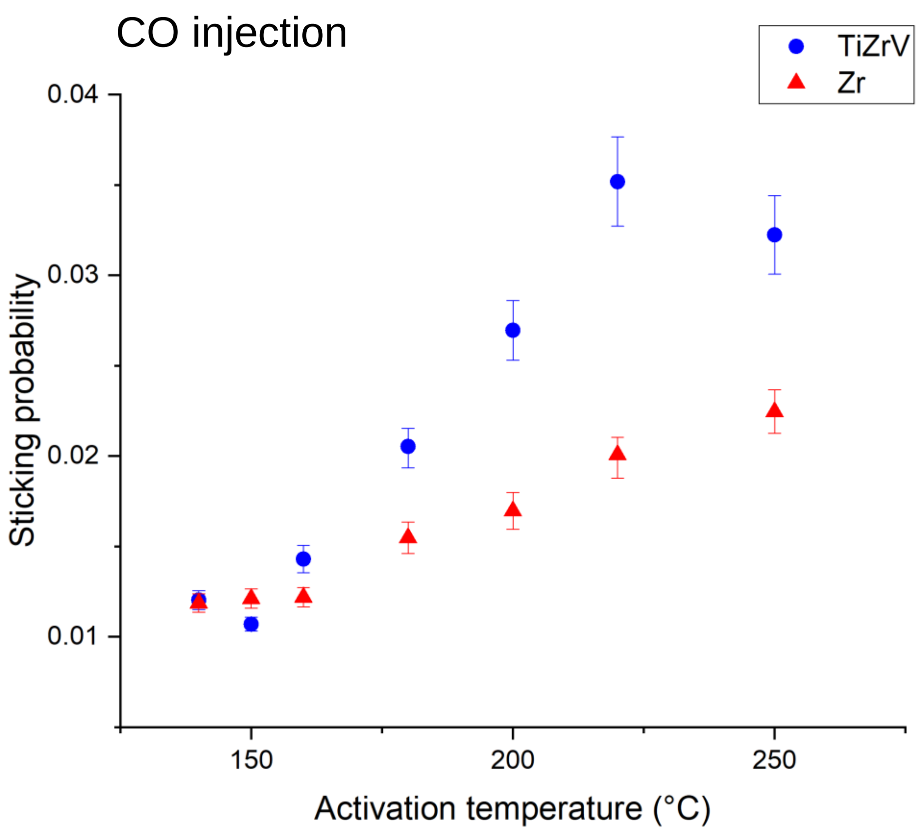
Vacuum tests – OFHC-Cu

Sticking probability and pumping capacity

- H_2 and CO sticking probabilities as well as CO pumping capacity (for T > 180 °C) were determined during the tests
- TiZrV demonstrated better sticking probability and pumping capacity

	TiZrV		Zr	
T_{act} (°C)	C_{CO} (CO/cm^2)	C_{CO} (ML)	C_{CO} (CO/cm^2)	C_{CO} (ML)
200	7.87×10^{14}	1.57	2.16×10^{14}	0.43
220	9.73×10^{14}	1.95	2.30×10^{14}	0.46
250	1.20×10^{15}	2.40	5.07×10^{14}	1.01



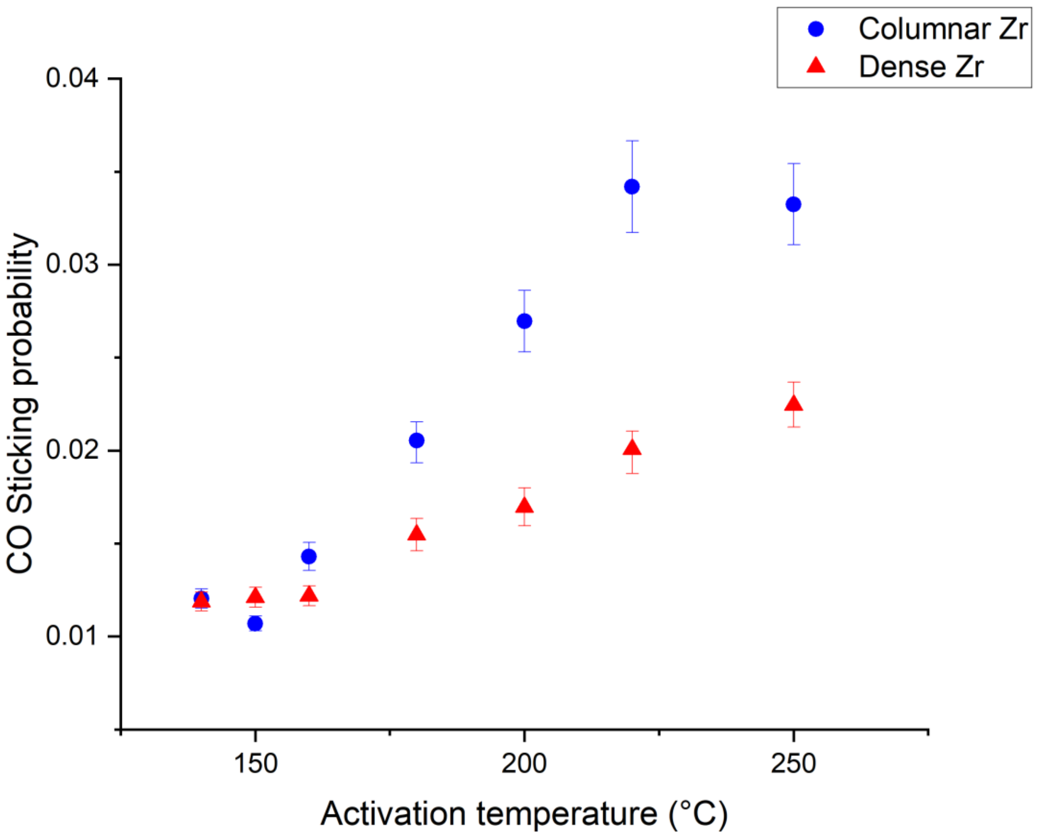


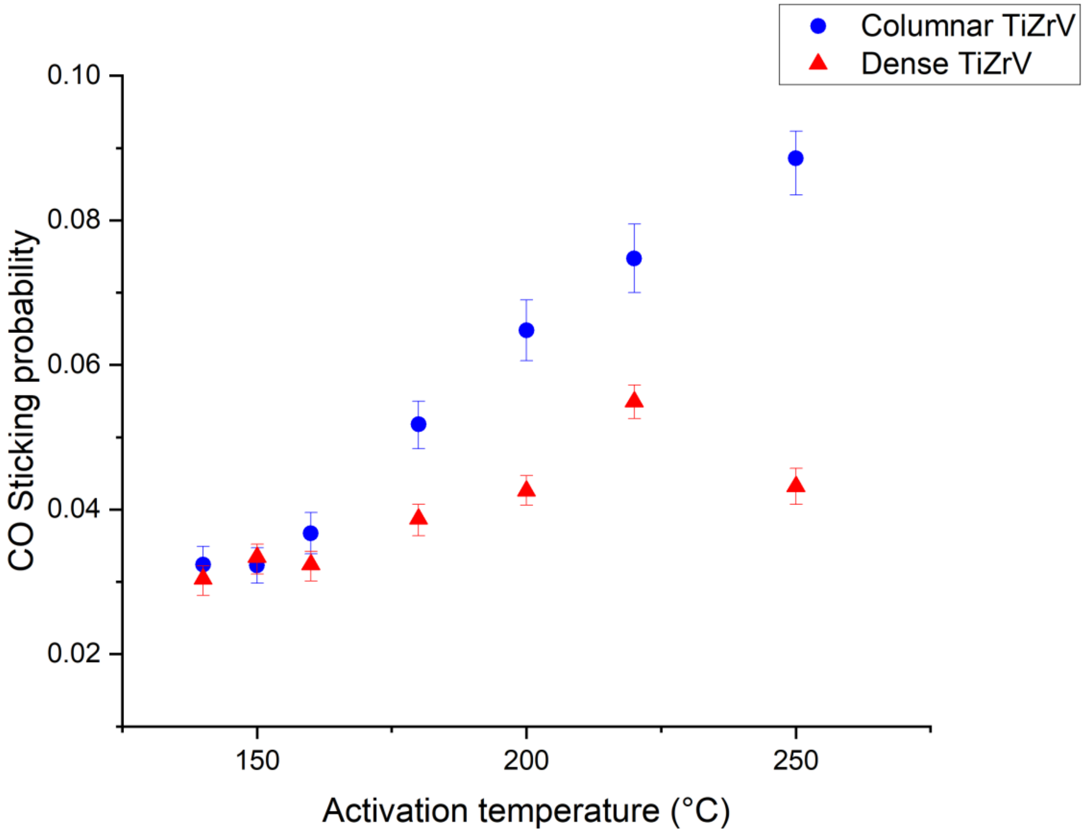
Vacuum tests – OFS-Cu

Sticking probability and pumping capacity

- CO sticking probability as well as CO pumping capacity (for T > 180 °C) were determined during the tests

	TiZrV				Zr			
	Dense		Columnar		Dense		Columnar	
T_{act} (°C)	C_{CO} (CO/cm^2)	C_{CO} (ML)	C_{CO} (CO/cm^2)	C_{CO} (ML)	C_{CO} (CO/cm^2)	C_{CO} (ML)	C_{CO} (CO/cm^2)	C_{CO} (ML)
180	5.34×10^{14}	1.07	7.74×10^{14}	1.55	4.29×10^{14}	0.86	5.28×10^{14}	1.06
200	6.72×10^{14}	1.34	6.98×10^{14}	1.4	4.38×10^{14}	0.88	7.87×10^{14}	1.57
220	8.75×10^{14}	1.75	8.32×10^{14}	1.66	6.56×10^{14}	1.31	7.92×10^{14}	1.58
250	1.10×10^{15}	2.20	1.80×10^{15}	3.6	8.61×10^{14}	1.72	1.20×10^{15}	2.4



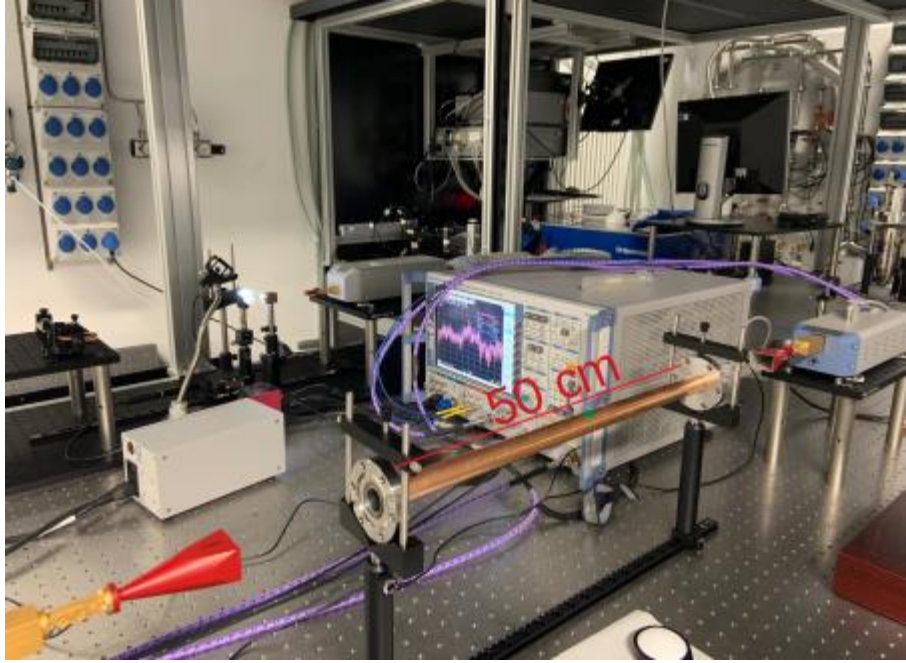


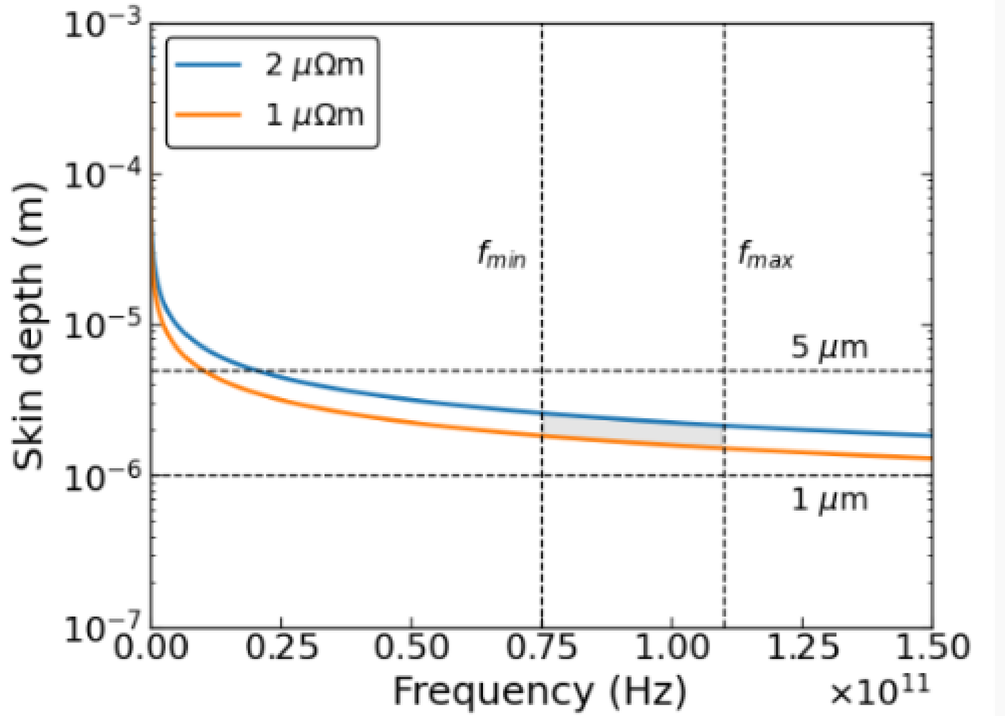
Resistivity measurements

Resistivity measurements

Conductivity of NEG coating

- Large number of chambers in PETRA IV system will produced out of OFS-Cu, which has a conductivity of around 60 MS/m
- However, NEG coating with its relatively poor conductivity (up to 0.8 MS/m for dense coating and down to 0.014 MS/M for columnar coating) might affect beam coupling impedance of coated vacuum chambers and wakefields, which may in turn induce collective instabilities
- Therefore, it is important to study how the NEG coating may influence the finite resistivity and potentially contribute to beam-induced wakes
- NEG conductivity depends on structure, chemical composition, preparation process, method used for magnetron sputtering etc.
- The coating has to be sufficiently thin in order to have a low impact on the resistive wall impedance
- Coatings with thickness of 1 µm are standard and expected in PETRA IV
- To estimate the conductivity of the NEG coating, thicker samples are needed
- Same conductivity is then assumed for thinner samples

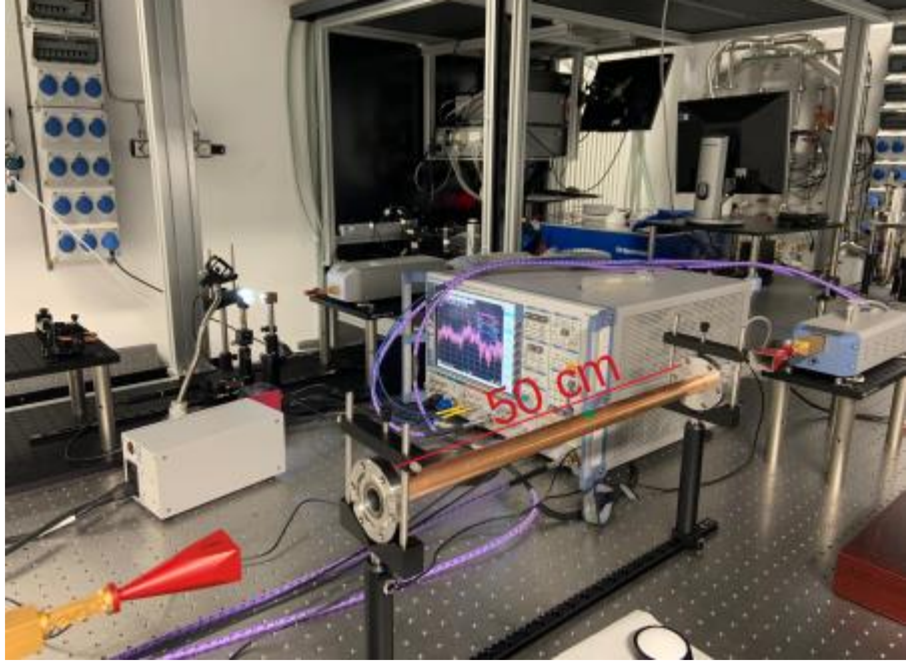


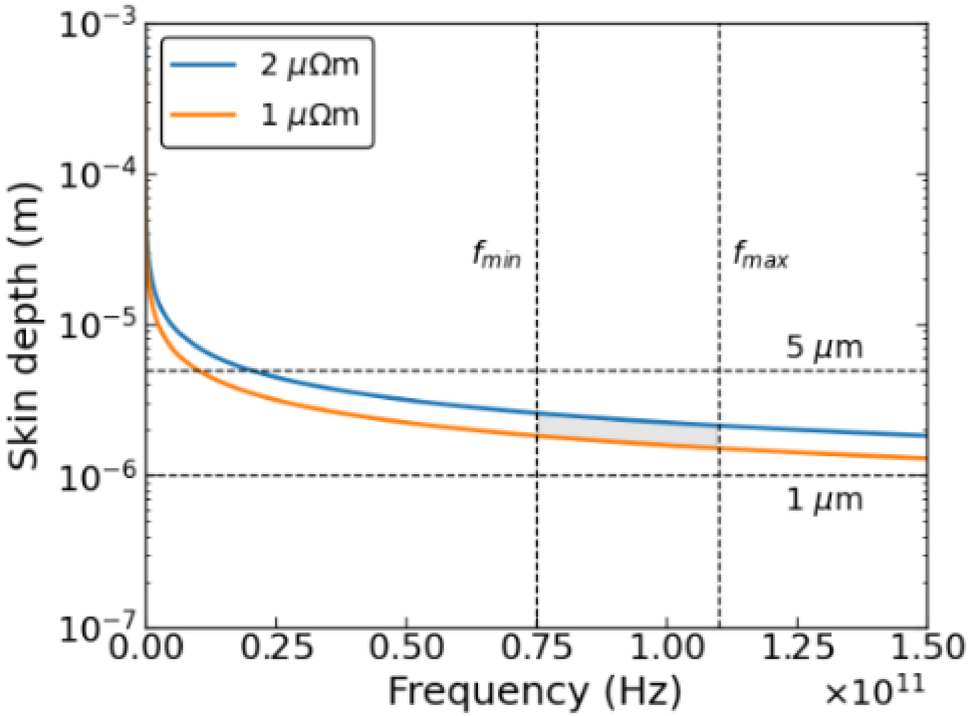


Resistivity measurements

System and measurement principle

- Microwave transmission method allows evaluating signal attenuation produced by the coating
- Measurement setup consists of:
  - Rohde & Schwarz vector network analyzer with two 75-110 GHz extension units: one for emitting and one for receiving the microwave signal
  - Two horn antennas
  - Set of holders for mounting the sample
- Attenuation of a microwave that is propagating in a circular waveguide can be expressed by $\alpha_{mn}^{TE}(f) = \frac{R_S}{Z_0 r\sqrt{1-\left(\frac{f_c}{f}\right)^2}} \times \left[\left(\frac{f_c}{f}\right)^2 + \frac{m^2}{({\chi'_{mn}}^2+m^2)}\right] Np/m$
- Material resistivity ρ can then be calculated using $R_s = \sqrt{\pi f \rho \mu_0}$
- For ρ to be accurate the skin depth has to be smaller than the coating thickness (coating can be considered as bulk)



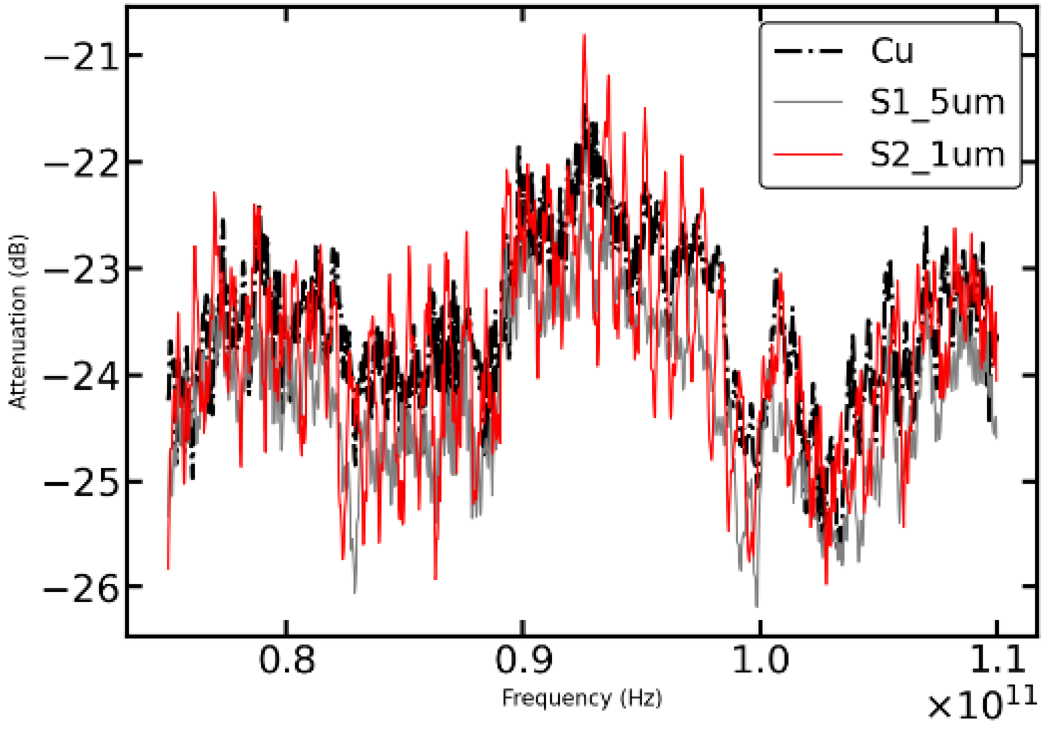


Resistivity measurements – OFHC-Cu

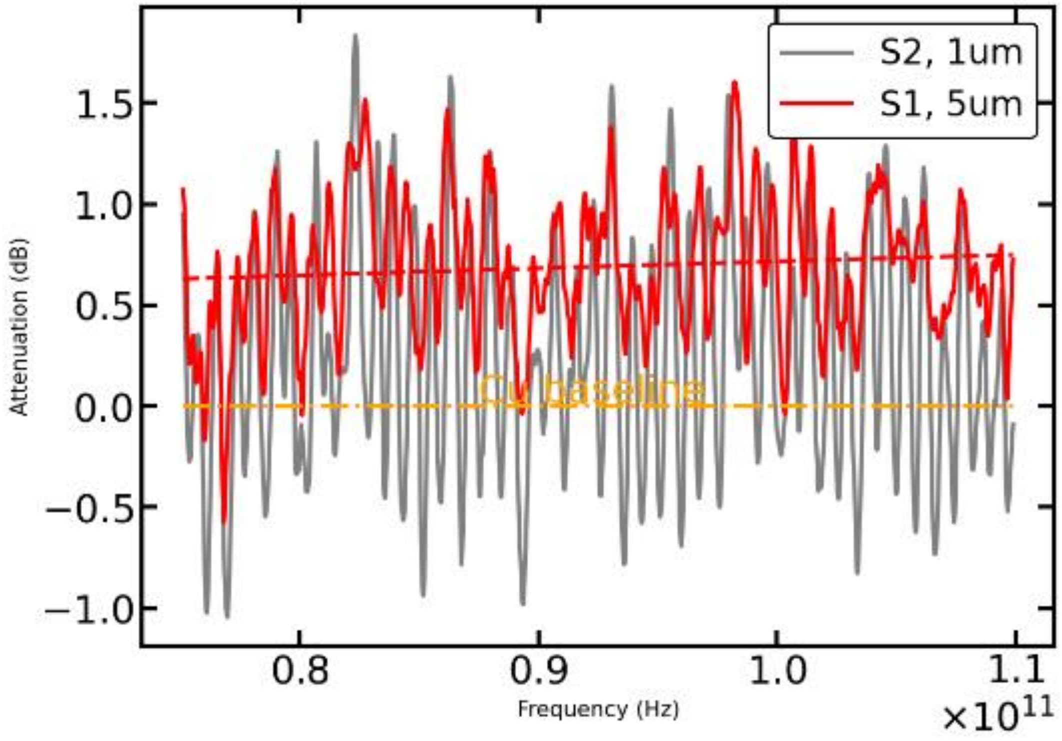
Samples 1 & 2: TiZrV coating

Estimated resistivity 5.4 µΩm, thin sample indistinguishable from bulk Cu

Raw data



Fitted data

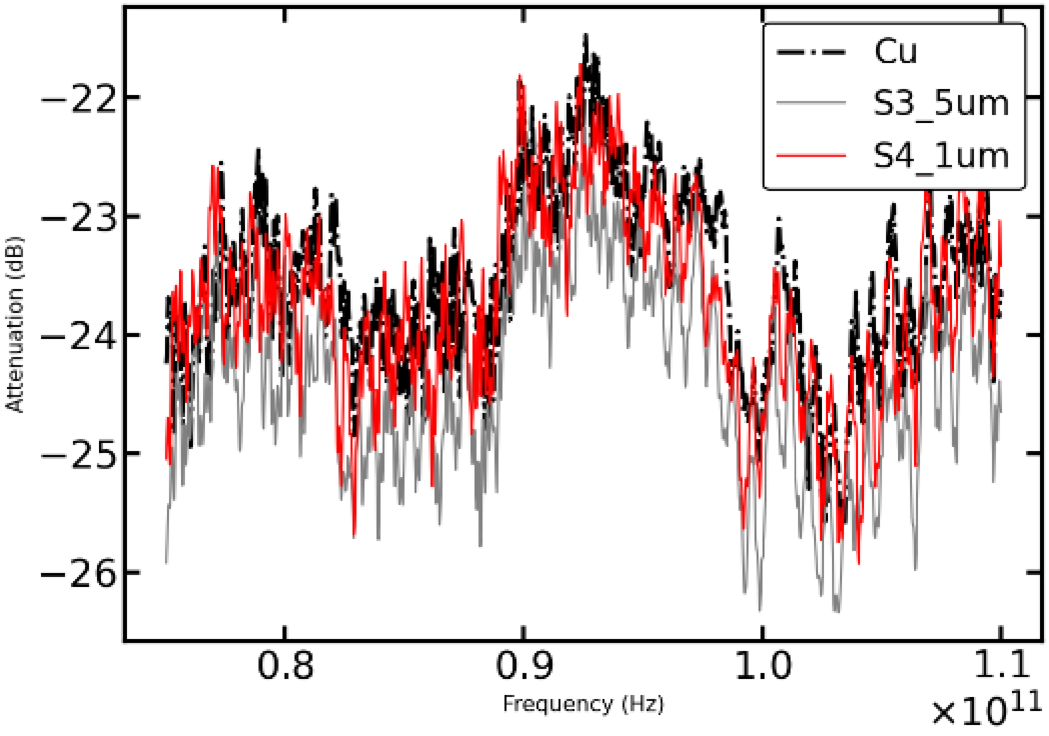


Resistivity measurements – OFHC-Cu

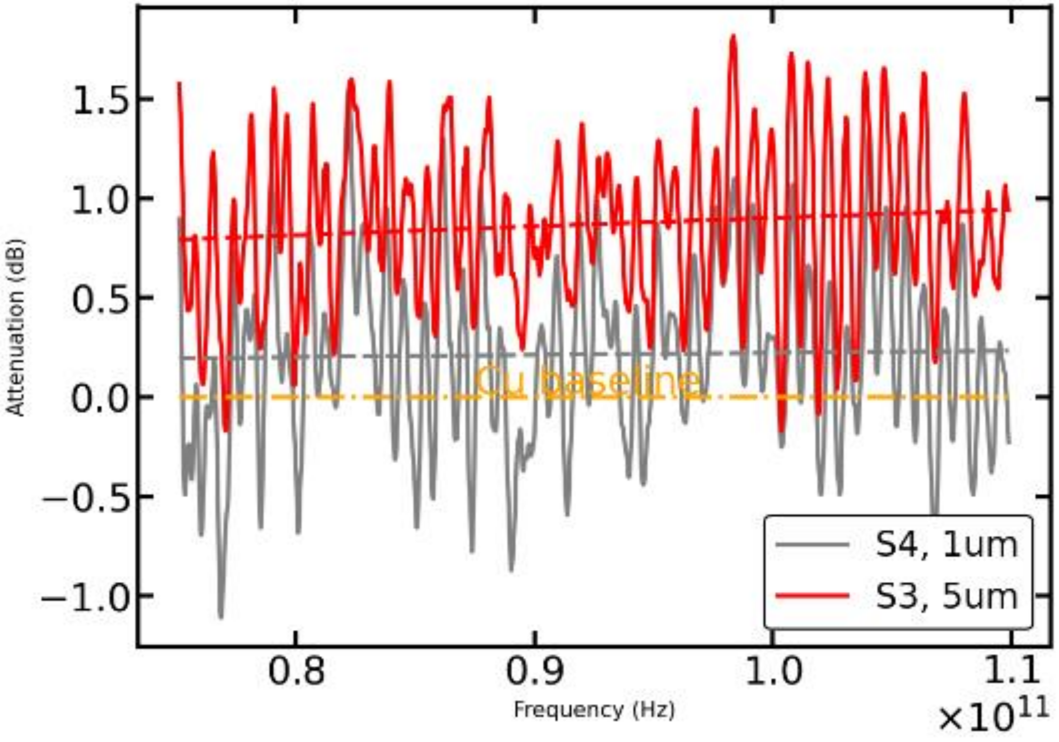
Samples 3 & 4: Zr coating

Estimated resistivity 8.5 μΩm, thin sample visible in the measurement

Raw data



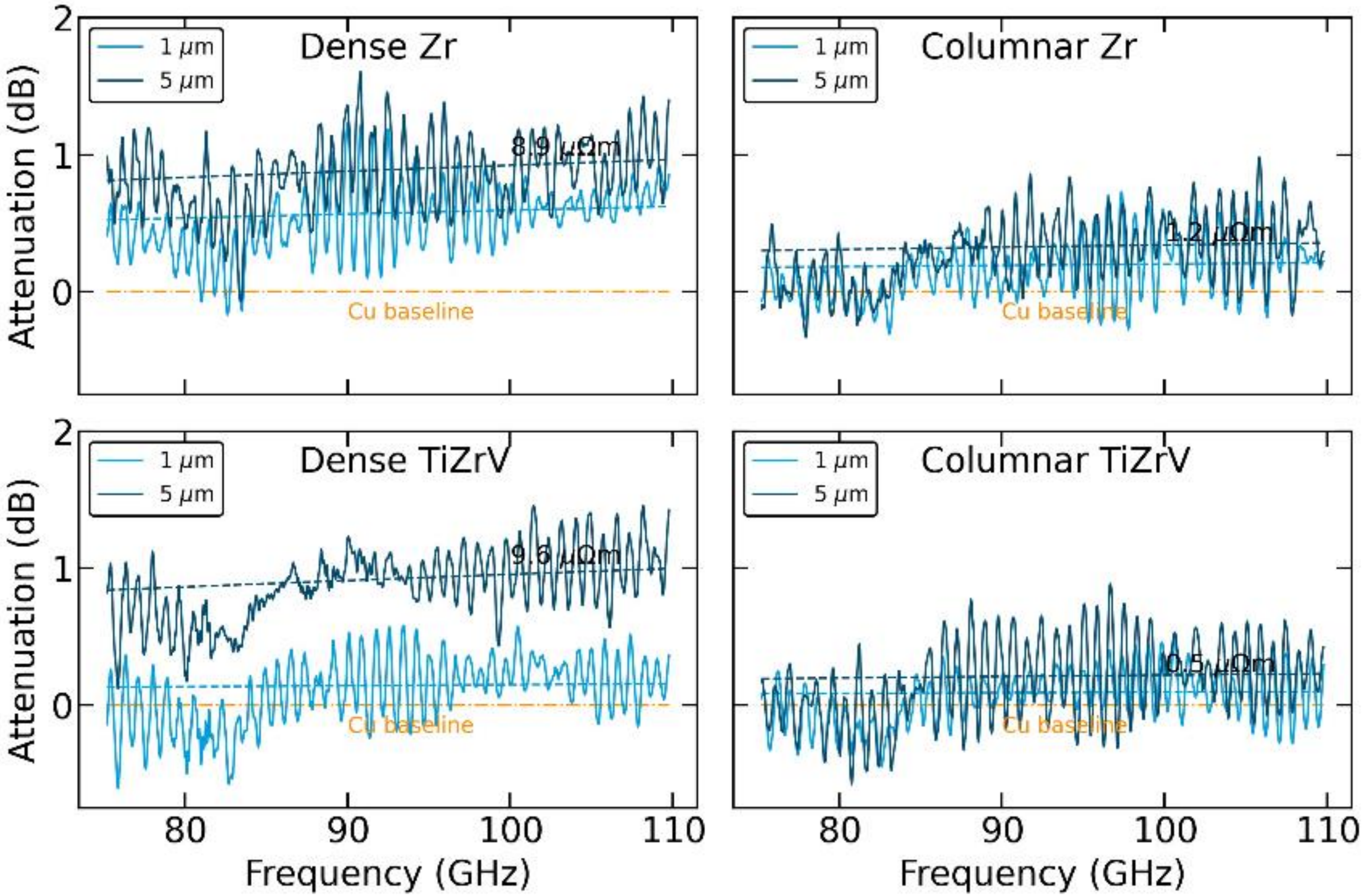
Fitted data



Resistivity measurements – OFS-Cu

Fitted data

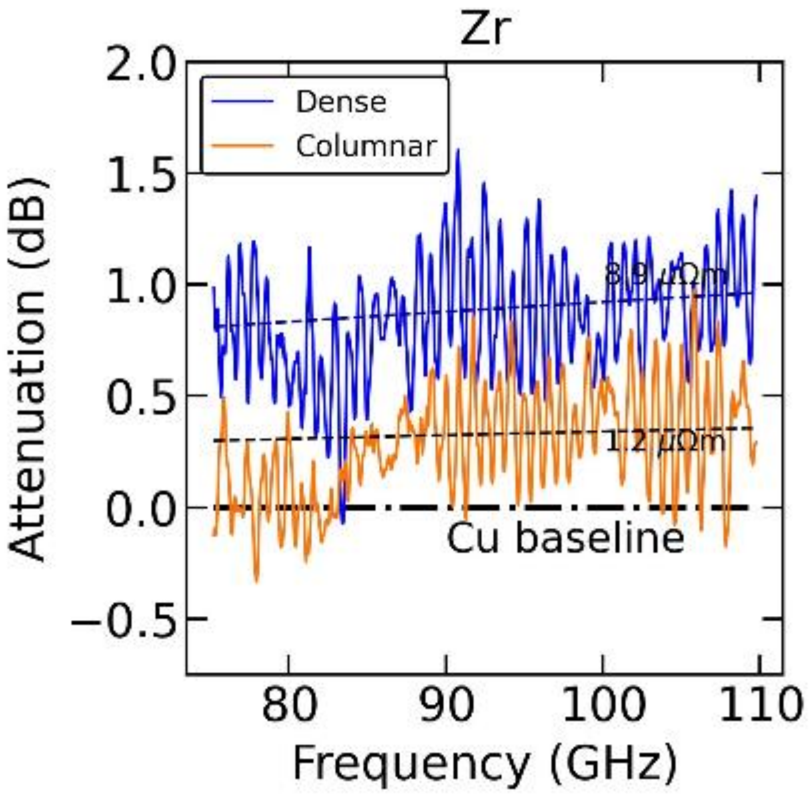
- Thin samples indistinguishable from the bulk copper
- The oscillations in frequency likely happen due to signal reflections at entrance and exit of the chamber
- The beating could be removed by tapered transitions from the waveguide ports to the pipe
- 10 measurements per sample done to reduce the impact of oscillations
- Data fitted to account for increasing attenuation with frequency

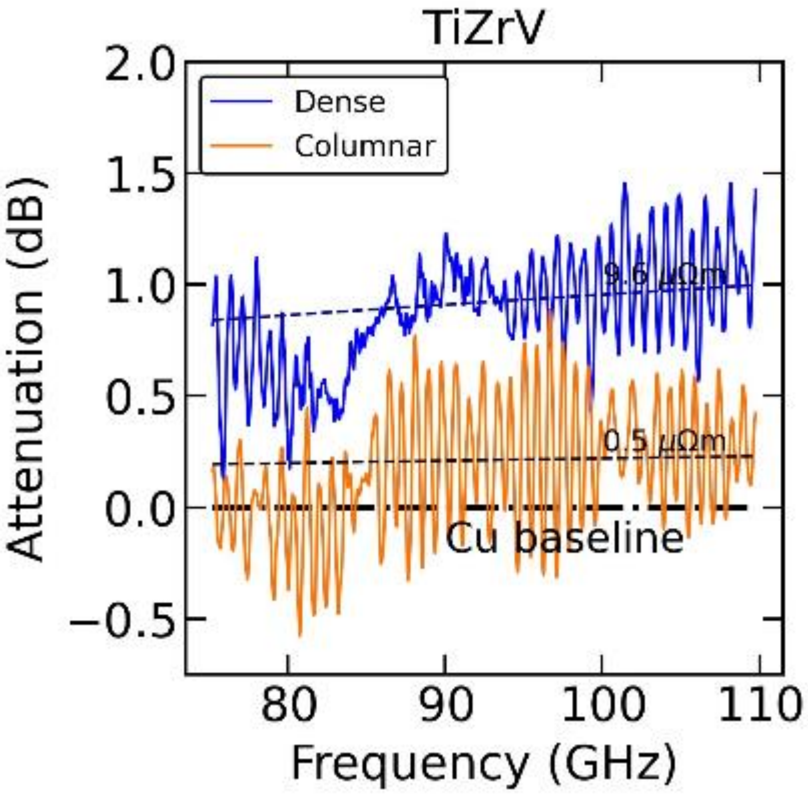


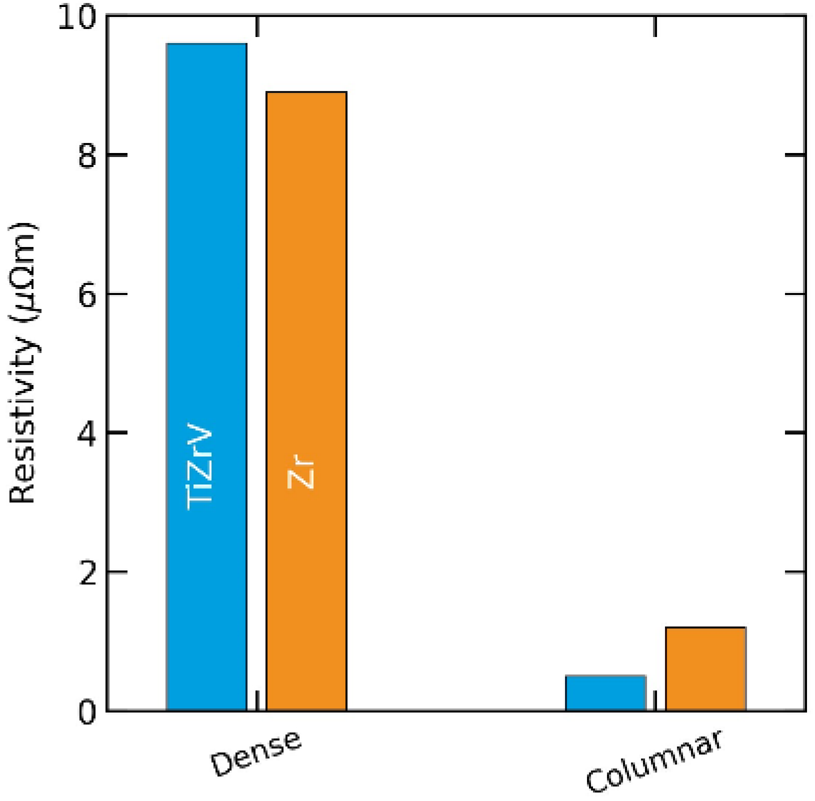
Resistivity measurements – OFS-Cu

Summary of the results for 5 µm samples

- Dense films show higher resistivity, dense TiZrV sample could be showing higher resistivity due to a surface defect
- The measured resistivity of 1 µm NEG films would not impact the PIV impedance significantly in a frequency range relevant to the 40 ps beam – anything less than 1 µΩm is accepted



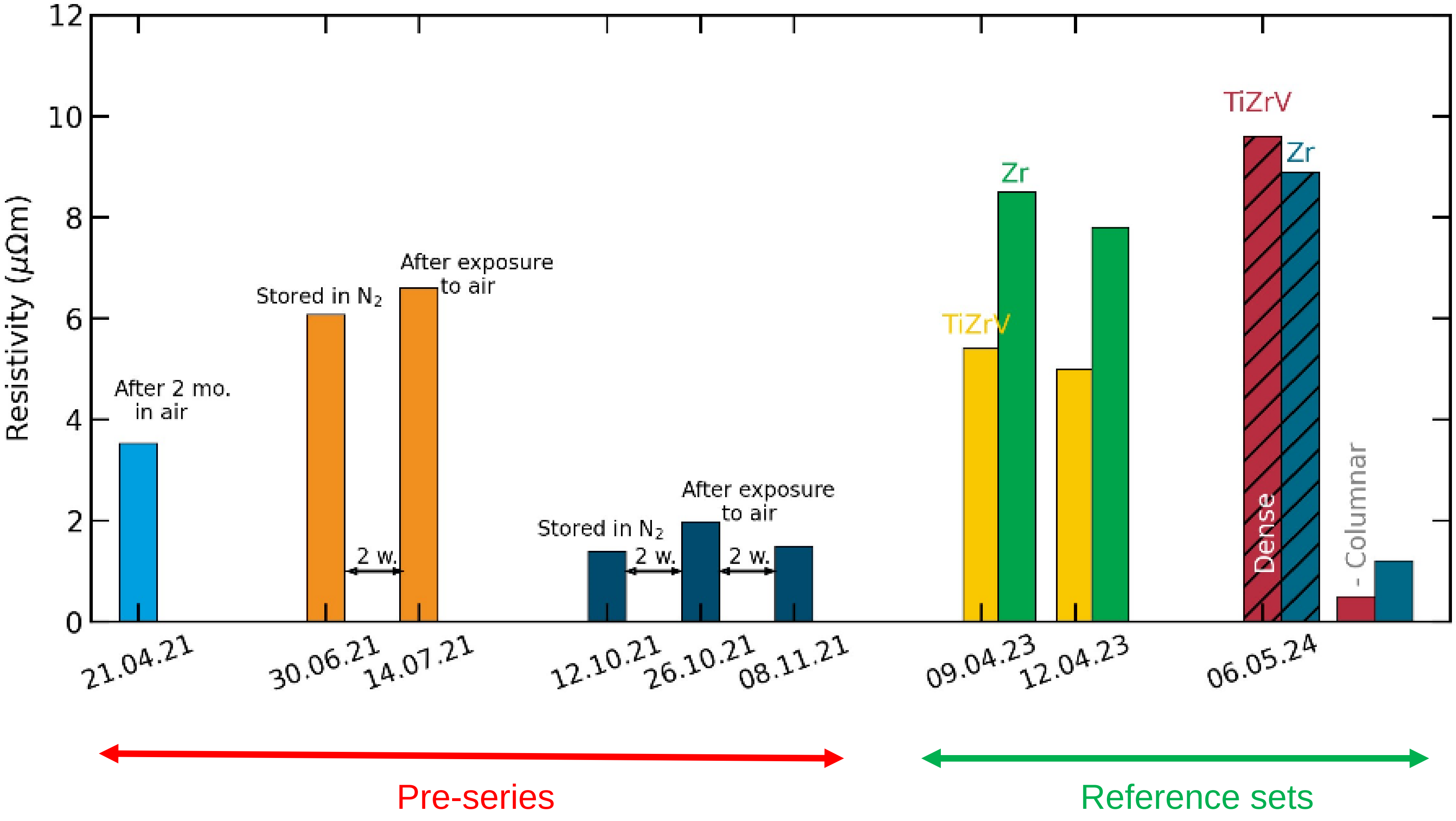




Resistivity measurements

Comparison with results obtained from OFHC-Cu

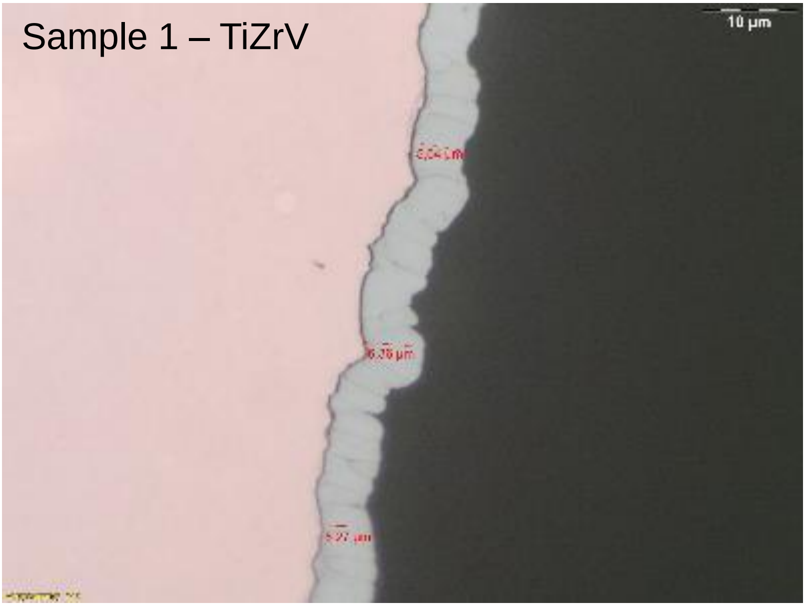
- More samples tested prior to assess how surface preparation and air exposure affect electrical properties
- Surface treatment before deposition affects the NEG resistivity
- Surface defects increase the resistivity (as seen on 1 µm and 5 µm dense TiZrV samples)
- Air exposure does not have an influence on electrical properties (neither NEG nor bulk Cu)
- All samples prior to the very last set were likely dense (all around 5 µm)

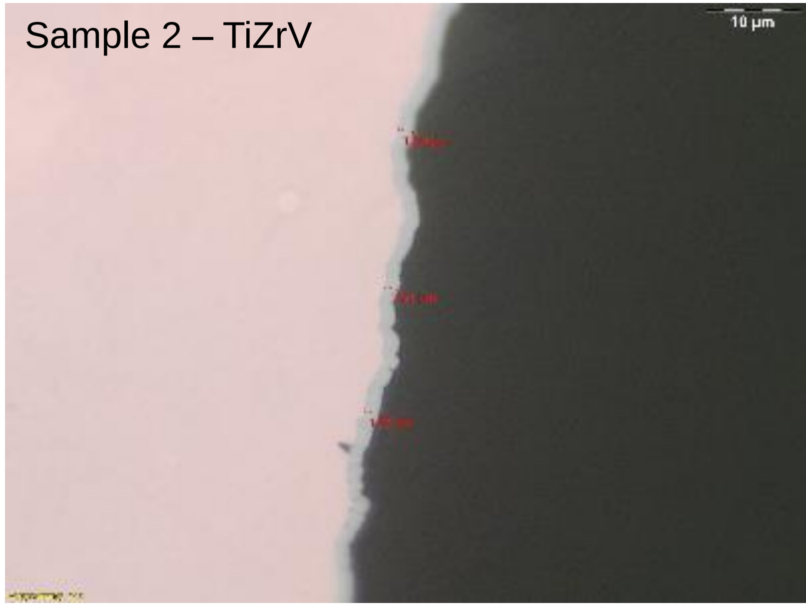


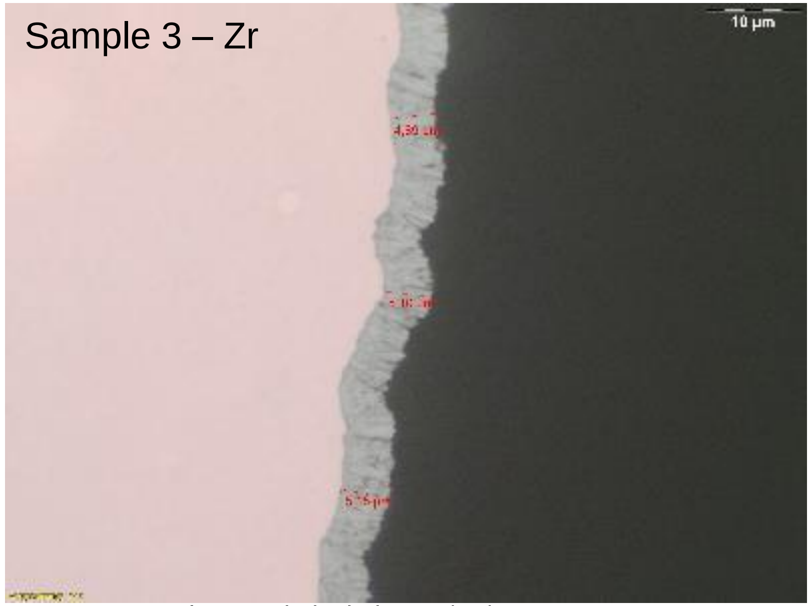
Surface characterisation

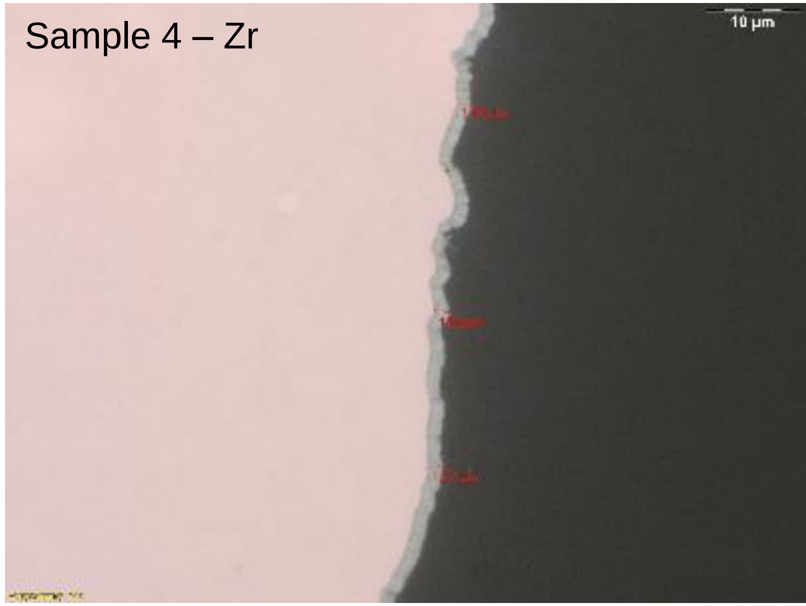
Thickness measurements

- 3 samples cut out of each tube; 3 locations tested on each sample
- Thickness varies depending on the location:
  - 5.2-7.5 µm and 1.3-2.3 µm for TiZrV
  - 4.5-5.8 µm and 1.3-2.2 µm for Zr
- Zr coating shows a more columnar structure
- Change from the expected thickness value is small enough to not influence the resistivity results (relevant for 1 µm coating)





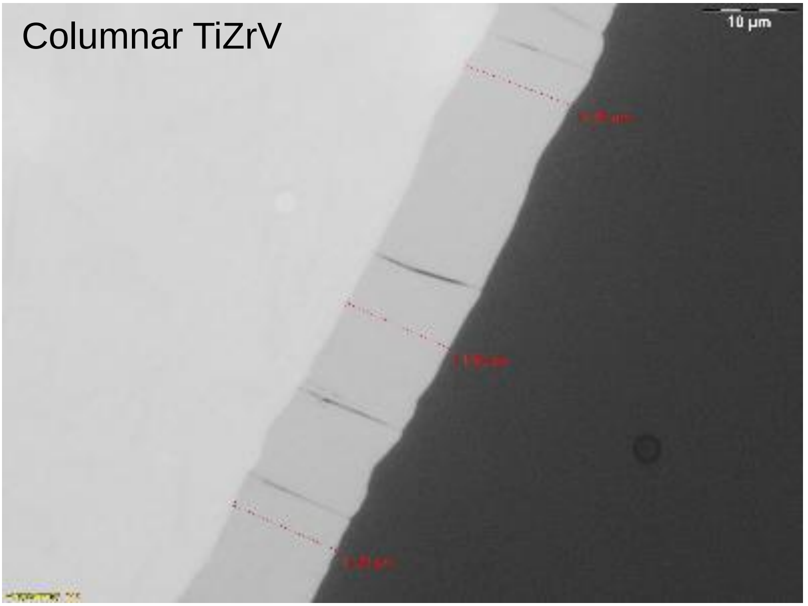


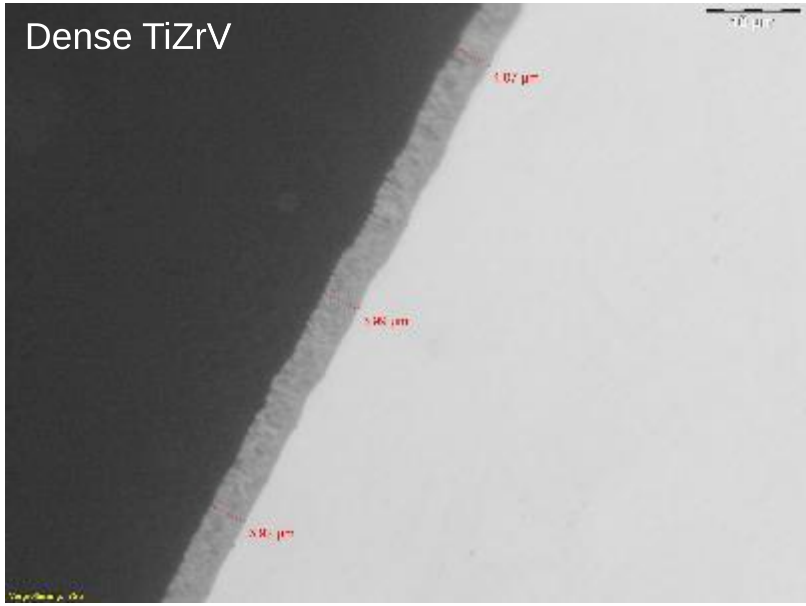


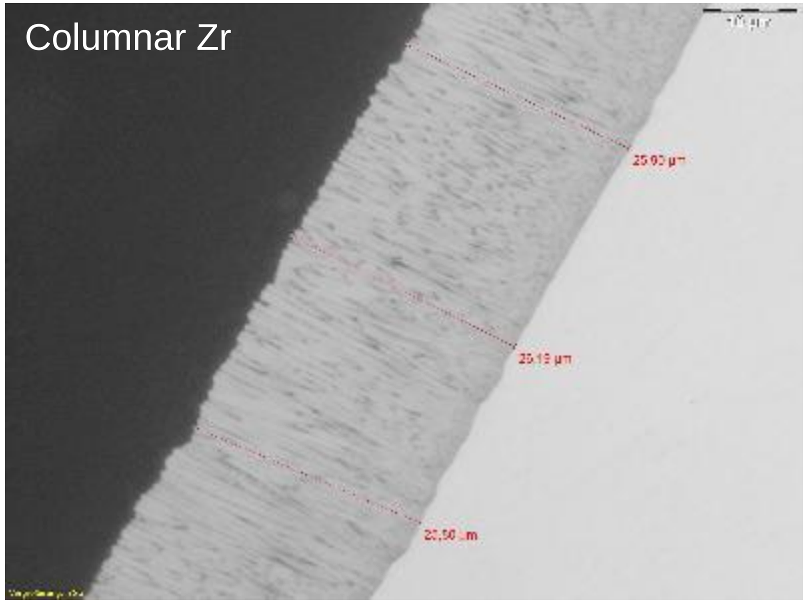
Surface characterisation

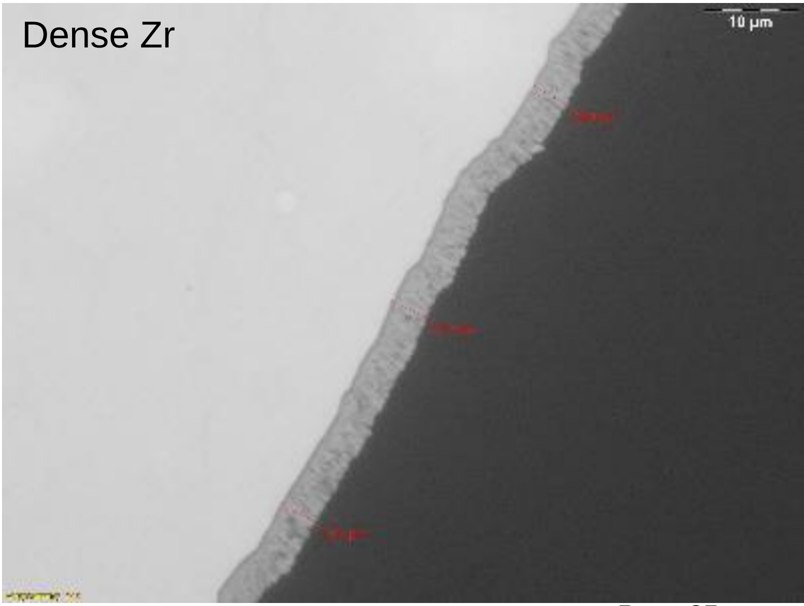
Thickness measurements

- 3 samples cut out of each tube; 3 locations tested on each sample
- *Thickness varies depending on the location:*
    - 0.6-1.1 µm and 2.1-4.1 µm for dense TiZrV
    - 1.6-4.3 µm and 6.7-12 µm for columnar TiZrV
    - 0.5-1.2 µm and 2.3-4.5 µm for dense Zr
    - 2.1-4 µm and 18.4-27.8 µm for columnar Zr
- Change from the expected thickness value is small enough to not influence the resistivity results (relevant for 1 µm coating)
- Dense samples should be coated for longer – the thickness is close to the skin depth









Undulator chamber dummy

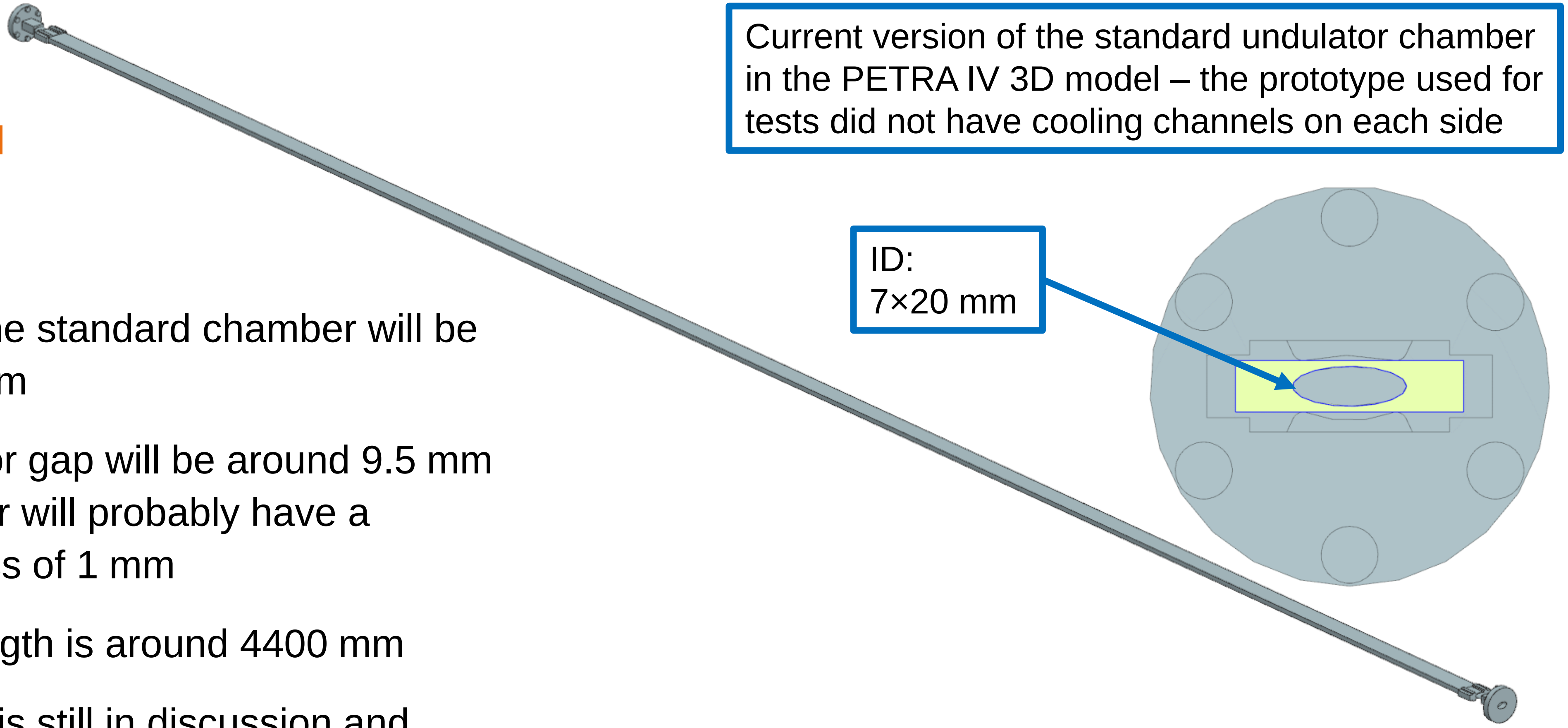
Motivation

PETRA IV Undulator Cell

Chamber geometry

- The cross section of the standard chamber will be an ellipse with 7x20 mm
- The available undulator gap will be around 9.5 mm (not less); the chamber will probably have a minimum wall thickness of 1 mm
- The current design length is around 4400 mm
- The undulator section is still in discussion and longer undulators are considered
- The material of the chamber will probably be an aluminum alloy (e.g. Al-6060)

Current version of the standard undulator chamber in the PETRA IV 3D model – the prototype used for tests did not have cooling channels on each side



Prototype dimensions:
Length = 5 m
OD = 16.3×7.5 mm
Wall thickness = 0.25 mm
Ellipse width = 7 mm
Material – 1.4404 (316L) Steel

Chamber preparation

Cleaning

Done after forming and welding the chamber

- ❑ Demineralised water rinsing (3 min, 0.3 µS/cm)
- ❑ **Protech 9** (30 min, 65 °C, 4 ml/l)
  - First 15 minutes in US bath (filled with Elma Clean at 65 °C)
- ❑ Demineralised water rinsing (2 min)
- ❑ N_2 flushing (1 min)
- ❑ Demineralised water rinsing (2 min)
- ❑ **Neodisher N** (5 min)
- ❑ Demineralised water rinsing (2 min)
- ❑ N_2 flushing (1 min)
- ❑ Demineralised water rinsing (2 min)
- ❑ Isopropanol rinsing (1 min)
- ❑ N_2 drying

Chamber preparation

Conditioning before bakeout

Cathode twisting

- 3×0.5 mm Ti, Zr, V wires twisted on site
  - Cleaning in a US bath beforehand

Installation

- Cathode inserted after chamber installation only
- Pumping carts connected to both sides of the chamber
- Frame for vertical support and centering with fixations every 25-30 cm
- Ceramic centering pieces and a weight (~270 g) attached to the cathode

Bakeout procedure

- System (along with the chamber) baked for 2.5 days at 200 °C

Deposition

Setup and parameters

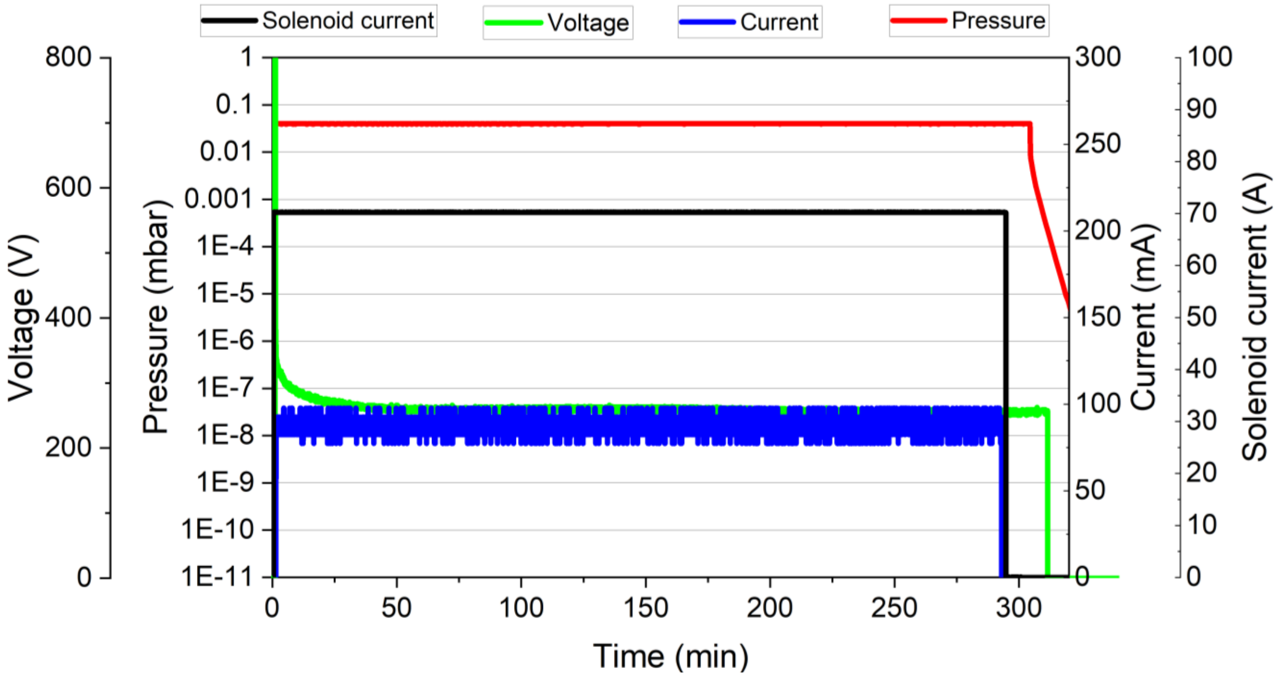
- Length of the solenoid – 1 m
- Coating done in 6 steps (from top to bottom)
  - Overlap between steps – 18 cm
  - Overlap at chamber ends – 5 cm

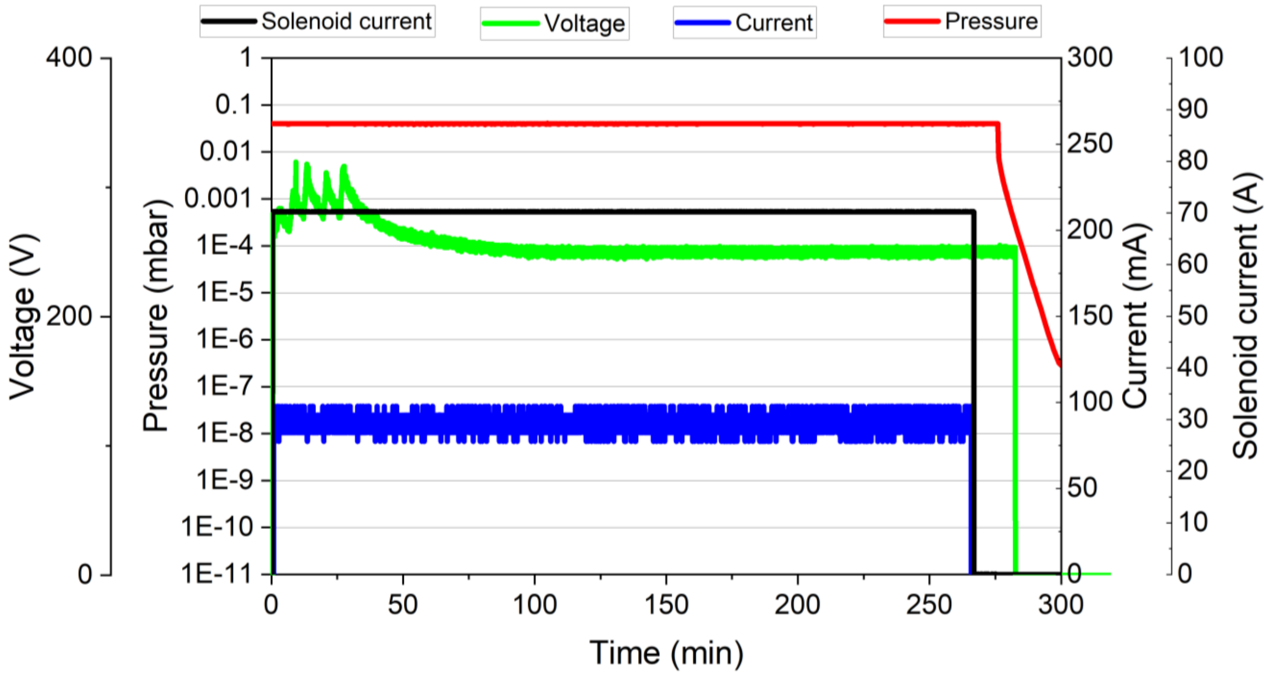
Coating parameters	
Current	80 mA
Power	21-23 W
Voltage	247-320 V
Discharge gas Kr pressure	0.02 mbar
Solenoid current	70 A
Coating time/step	1-292 min
Total coating time/step	282-335 min

Deposition

Attempts

- Middle sections (3 and 4) most problematic
  - Many alignments and adjustments required
  - Alignment precision of ±0.2 mm along the entire chamber
- Countless attempts to start and maintain discharge
- Solution: starting the process at the nearest position possible, then moving the magnet to the desired point
- Coating for around 300 min at each step in total (multiple starts)
  - Set in order to get coating thickness of approximately 1 µm



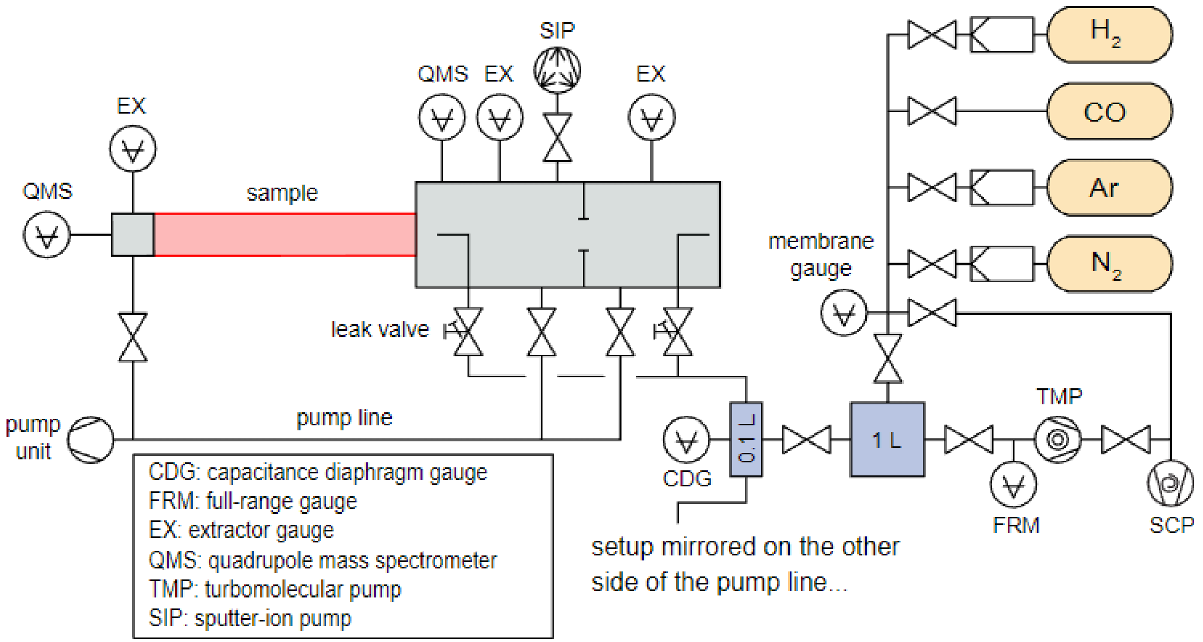


Vacuum tests

Preparations

Pumping Speed Measurement (PSM) setup

- No extractor gauge on the left side of the sample – quadrupole mass spectrometers used instead



Bakeout/Activation

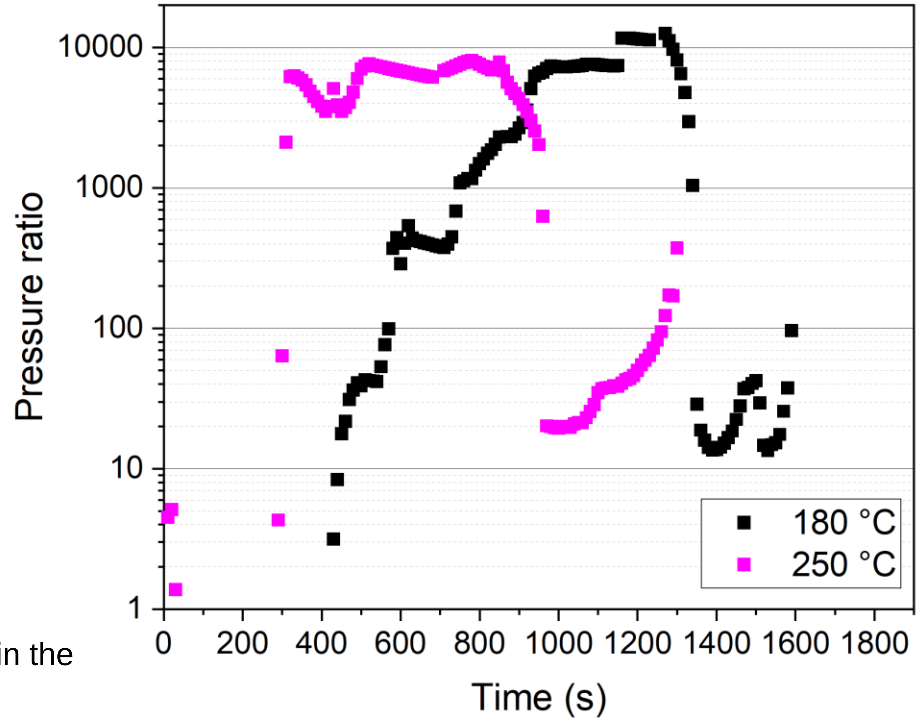
- Pumping both ends of the chamber
- Standard STFC ASTeC bakeout/activation procedure applied
  - Chamber kept at 80 °C with the rest of the system at 200 °C
  - System kept at 150 °C for two hours at the start of sample activation
- **Two activations**: 180 °C (right after the bakeout), 250 °C (after injections that followed the first activation)

Sticking probability tests

H_2 injections

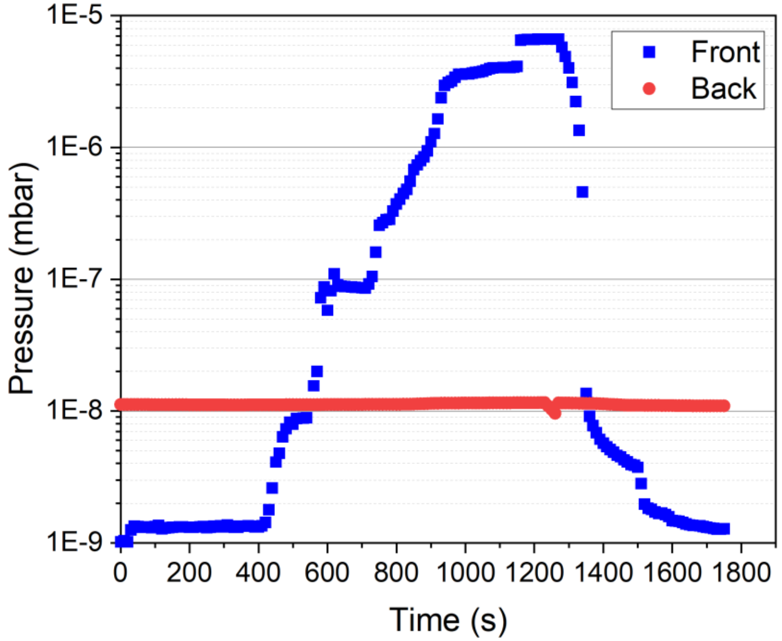
Description

- Turbo pump open
- Short injections (5 min after observing increased pressure at the end of the sample; over 10 min in total)

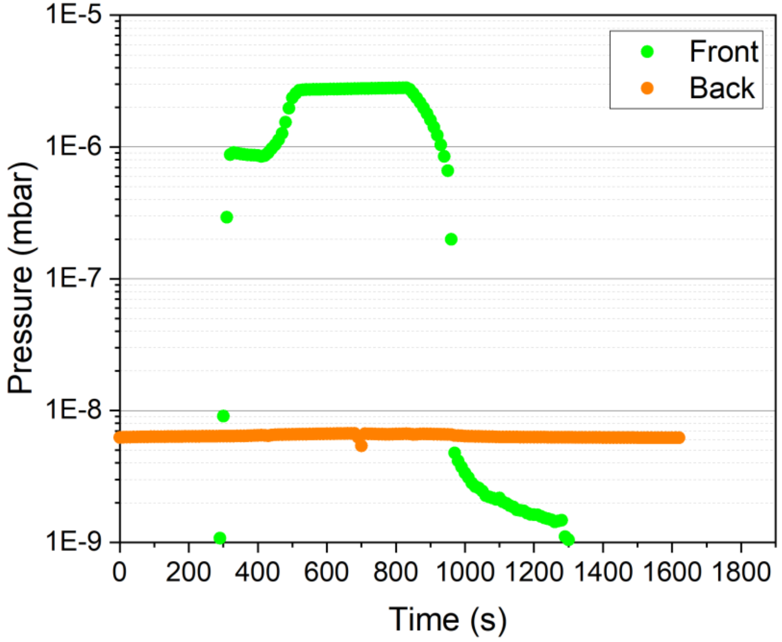


*background subtracted in the ratio graph

180 °C



250 °C

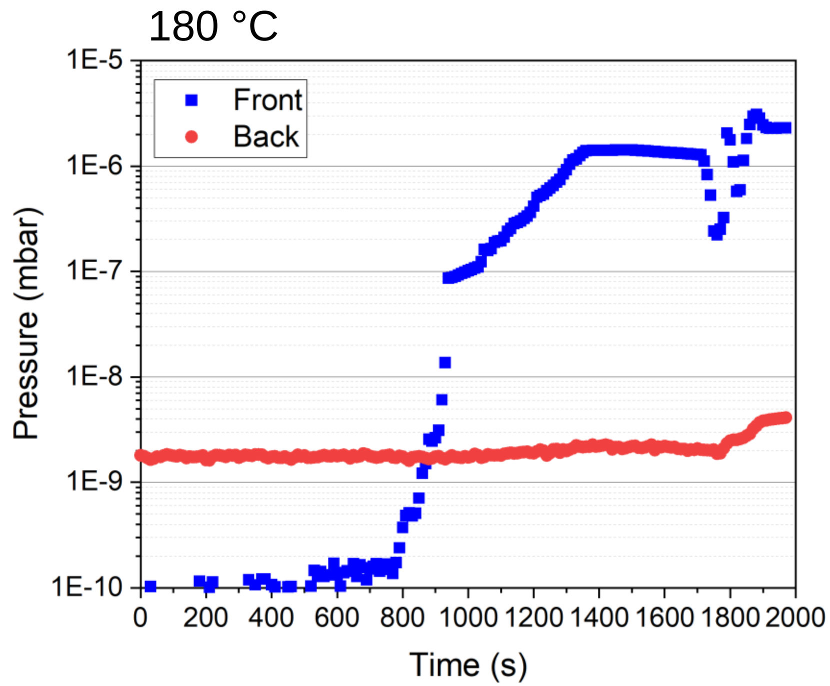


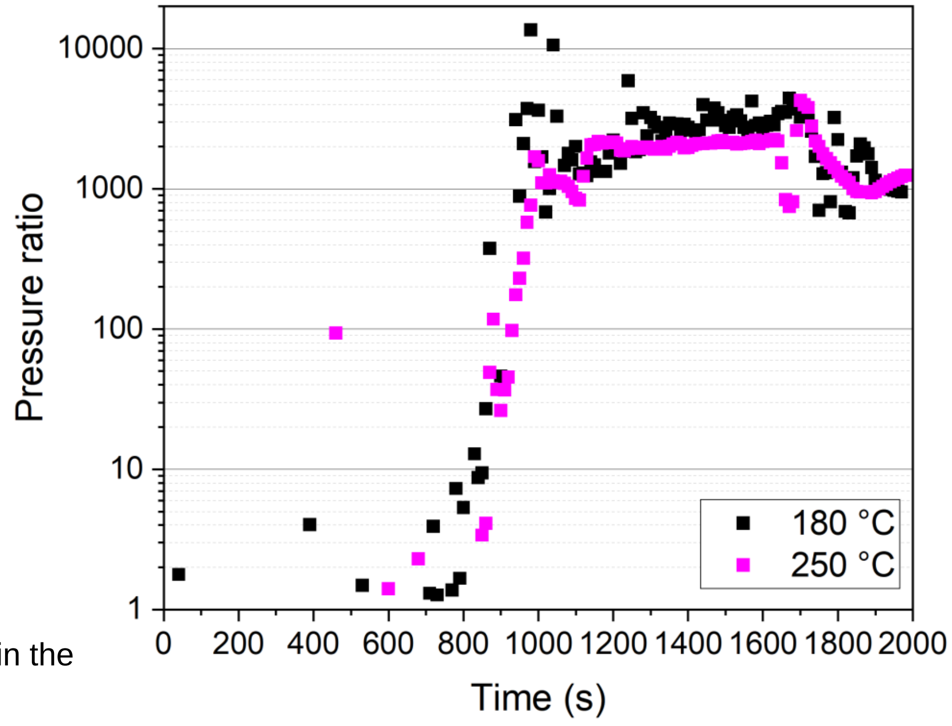
Sticking probability tests

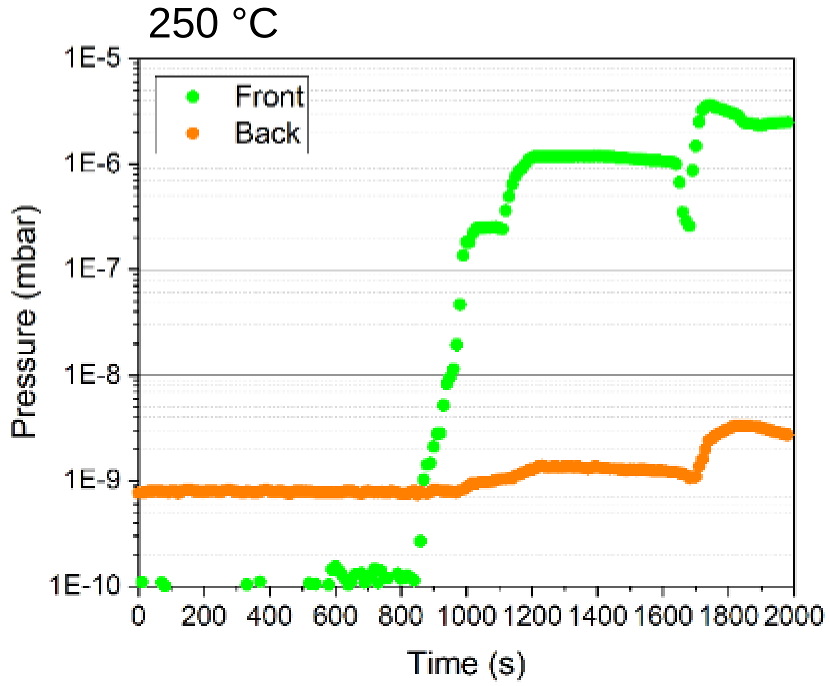
CO injections

Description

- Turbo pump kept open to have a better background pressure
- Short initial injection (5 min after observing increased pressure at the end of the sample), leak rate reduced before closing TMP







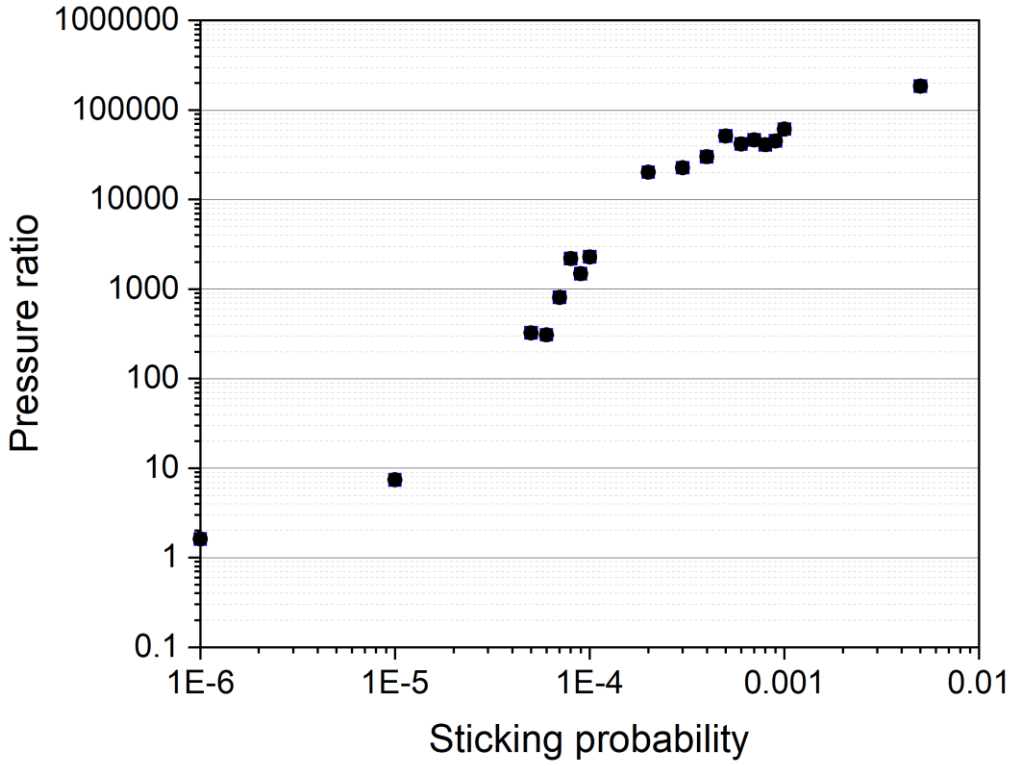
*background subtracted in the ratio graph

Further tests

Capacity measurements

Molflow+ model

- Sticking probabilities from 10^{-6} to $5x10^{-2}$ applied to the entire chamber inner surface area



Capacity tests

- Should be a more accurate method to evaluate the pumping
- Turbo pump closed right after initial CO injection, gauges switched off later
- Injections continued overnight to saturate the sample (20-24 hours)
- Final $P_{front}/P_{end} < 10$

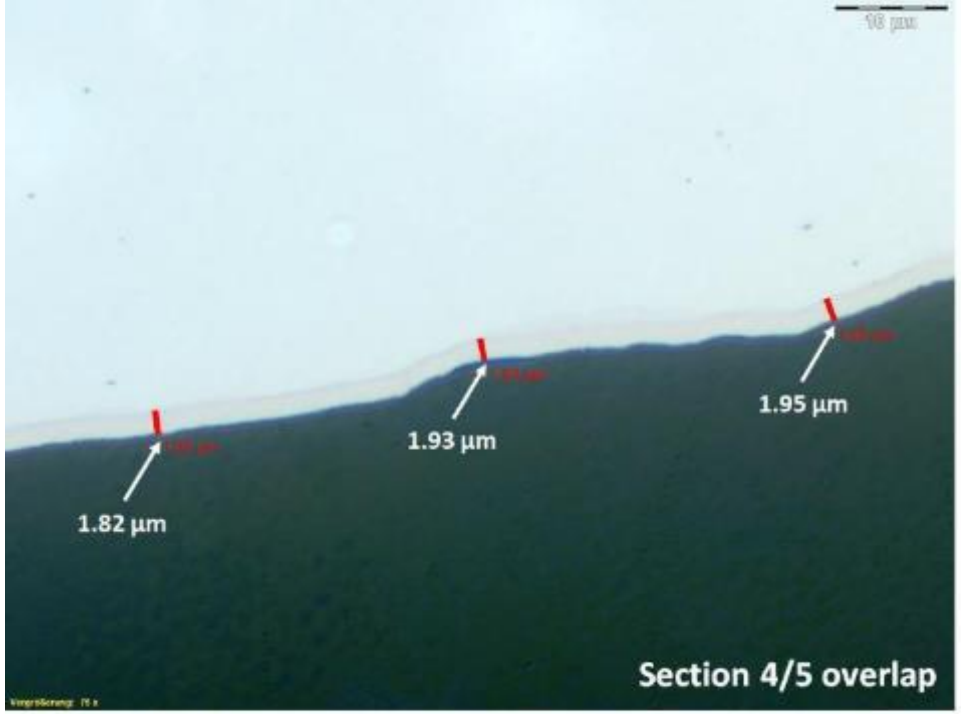
T_{Act}	α_{H2}	α_{CO}	C (ML)	C (CO/cm^2)
180 °C	$1x10^{-4}$	$1x10^{-4}$	2.79	$1.4x10^{15}$
250 °C	$2x10^{-4}$	$9x10^{-3}$	4.00	$2.0x10^{15}$

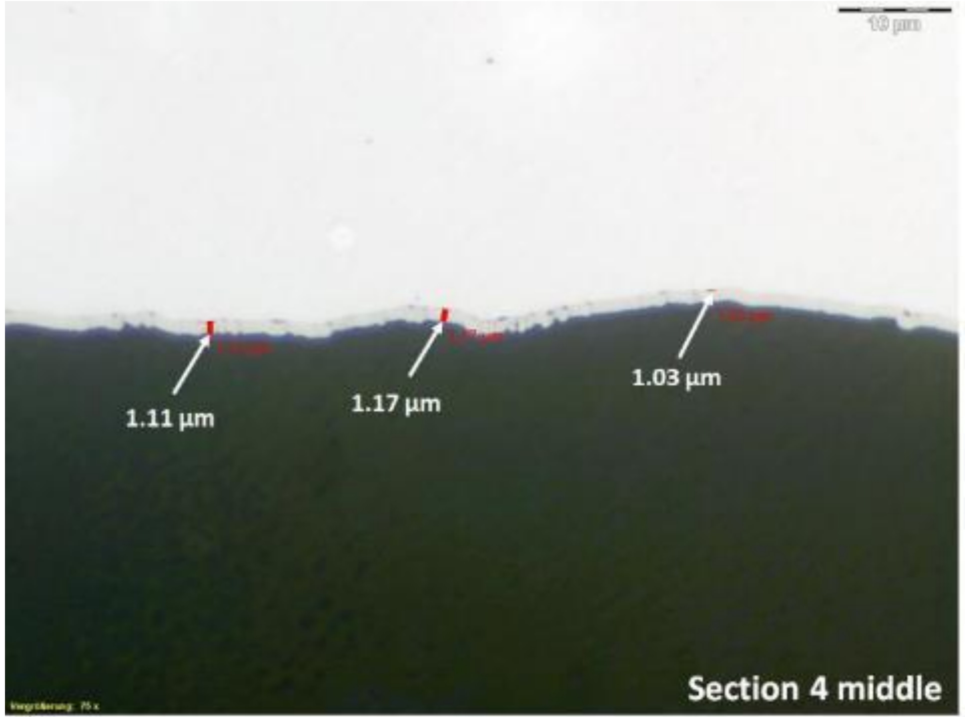
Surface characterisation

Thickness measurements

Laser scanning microscope analysis

- 15 locations analysed
  - 3 section overlaps (doubled coating time)
  - 2 middle sections
- Samples embedded at 80 °C (no pressing force applied)
- As expected, thicker coating seen where the sections overlap
- Coating fractured in Section 4 (multiple attempts at coating)
- Stable coating process resulted in a more uniform film





Challenges

In various stages of tests

Deposition

- Cumbersome cathode/chamber installation
- Centering is extremely important for such narrow chambers; fixing a weight to the cathode is essential
- Precise vertical alignment needed – chamber should be as straight as possible after production
- Thermal expansion of the cathode and chamber has to be taken into consideration when installing centering pieces and baking, respectively

Pumping tests

- Some complications due to low transmission both with tests and simulations
  - Difficult to see pressure increase at the end of the chamber
  - Takes a long time to saturate (device protection has to be reviewed)
- Molflow+ model should include transverse facets along the tube to allow for a case of chamber saturation at the front
  - Will be done when the geometry is (close to) final

Considerations for future samples

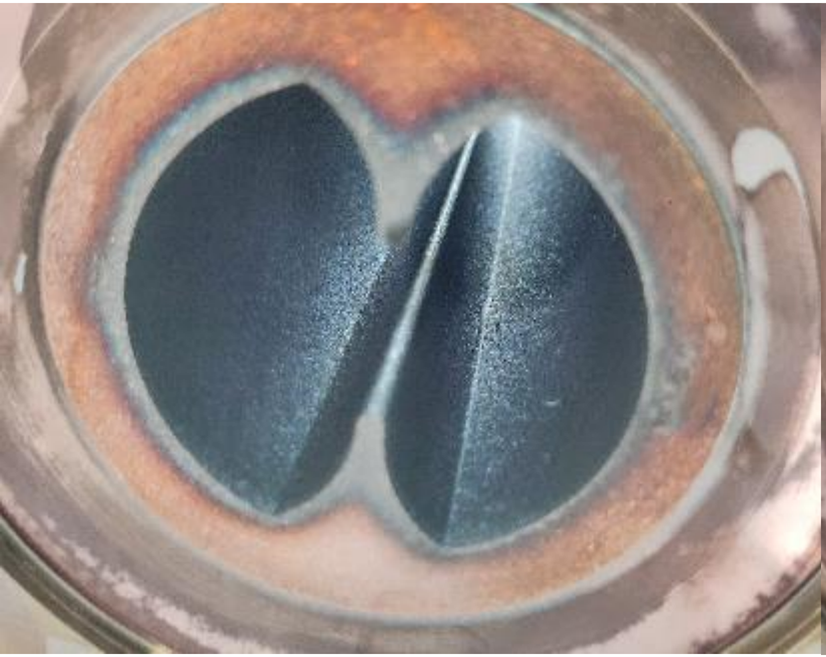
- Chamber geometry will be changed to resemble the actual case more
  - Shorter with same ellipse eccentricity
- Alternative coating could be tried (e.g. pure Zr)
  - Allows for a single metal wire cathode – easier to prepare and handle, especially if 1 mm Zr wire is used instead of 0.5 mm Ti, Zr and V wires
- Cathode conditioning in a separate chamber prior to the deposition
  - Short sputtering before the first use

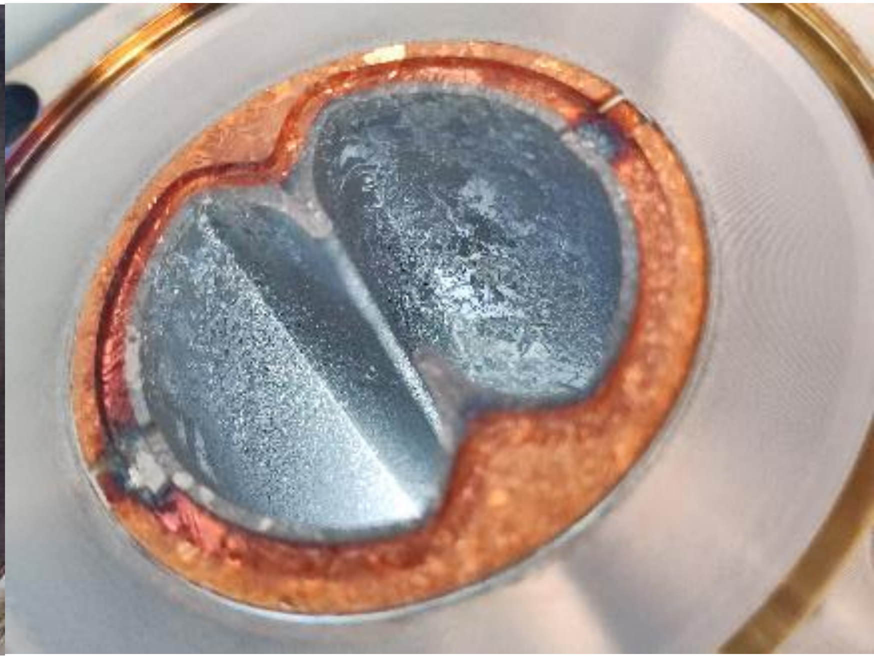
Coating tapers

- Material: Taper 1 - CW022A (99.98% Cu), Taper 2 - CW021A (99.95% Cu)
- Taper 2 was sent for production at an external company, wire eroded after brazing
- Both tapers brazed at 850 °C
- Taper 1 wire eroded before brazing
- Challenges with deposition:
  - Thickness would not be uniform due to the irregular chamber geometry
  - Fenders might not get coated as they are shadowed (Shielding?)
  - Fender tips will be coated most (closest to the cathode); delamination possible
- Sputtering time increased to 8 hours

Coating tapers

- Tapers fully coated, as well as fenders
- Thickness not known yet
- As long as the cathode is centered, there should not be a problem with fender coating





NEG ageing tests

Test description

- 4 samples (1 m long, 20 mm ID) coated for testing

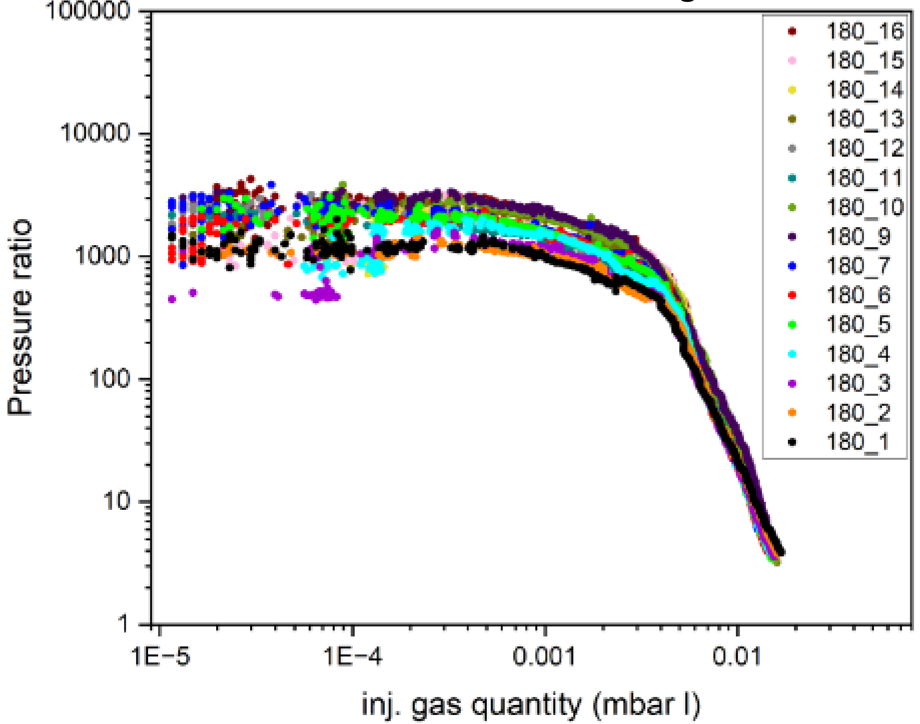
Sample	Target	Etching	Deposition mode	Coating	Thickness (µm)	Set parameter	t (h)	p (mbar)	$I_{solenoid}$ (A)
TK Ag 24	TiZrV	Y	DC (AEPP)	Columnar	1	55 W	5	4.8e-1	70
TK Ag 25	TiZrV	Y	Pulsed DC (AEPP)	Dense	1	70 W	5	7.2e-2	70
TK Ag 27	Zr	Y	Pulsed DC (AEPP)	Dense	1	70 W	5	7.2e-2	70
TK Ag 29	Zr	Y	DC (AEPP)	Columnar	1	55 W	6	4.8e-1	70

- Zr samples (dense and columnar) tested first
- Starting temperature – 180 °C; increase to 200 °C once the pumping properties see stable degradation
- Three-day activation to be done at the end to check if sticking probability recovers

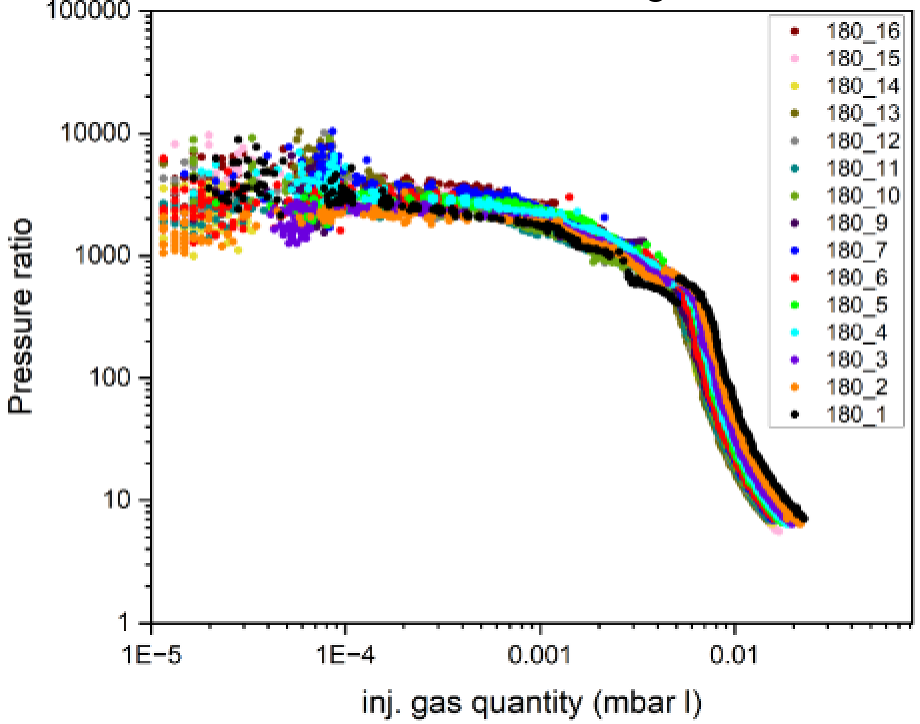
CO pumping – activations to 180 °C

- 15 activations at 180 °C degrees (the eighth activation is not taken into account)
- For columnar coating, after the 9th activation, there was no visible increase in pressure ratio
- For dense coating, until the last activation, slight increases in pressure ratio was visible

Columnar coating



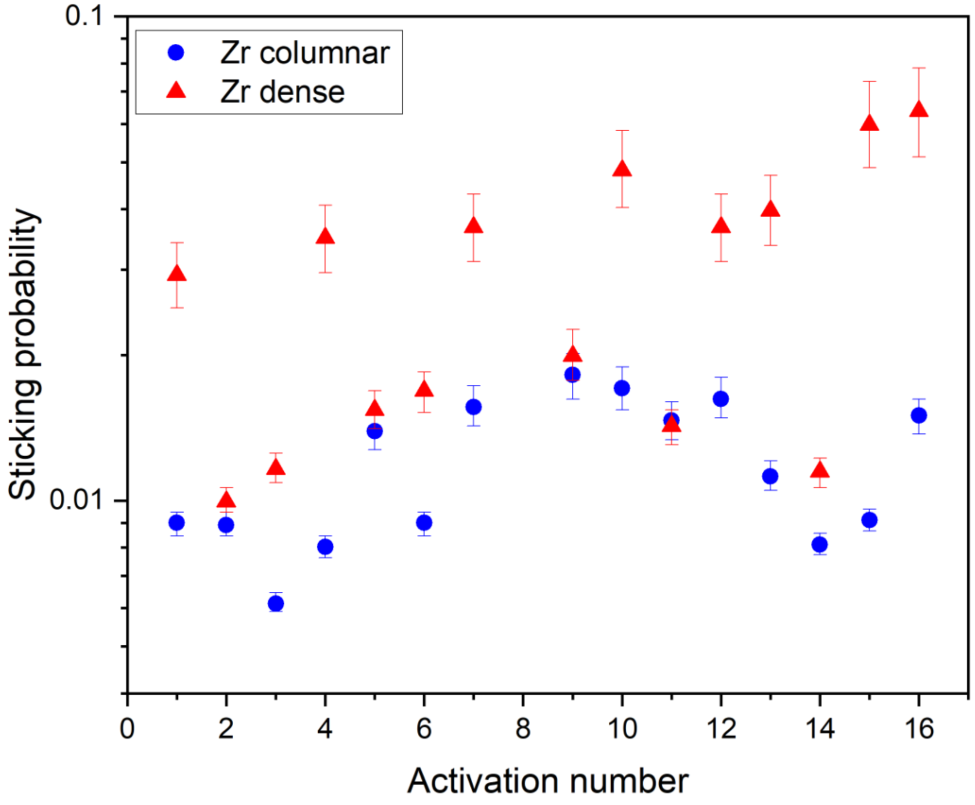
Dense coating



CO pumping – activations to 180 °C

CO sticking probability and pumping capacity

- Increasing sticking probability for the dense coating
- Slow reduction in capacity

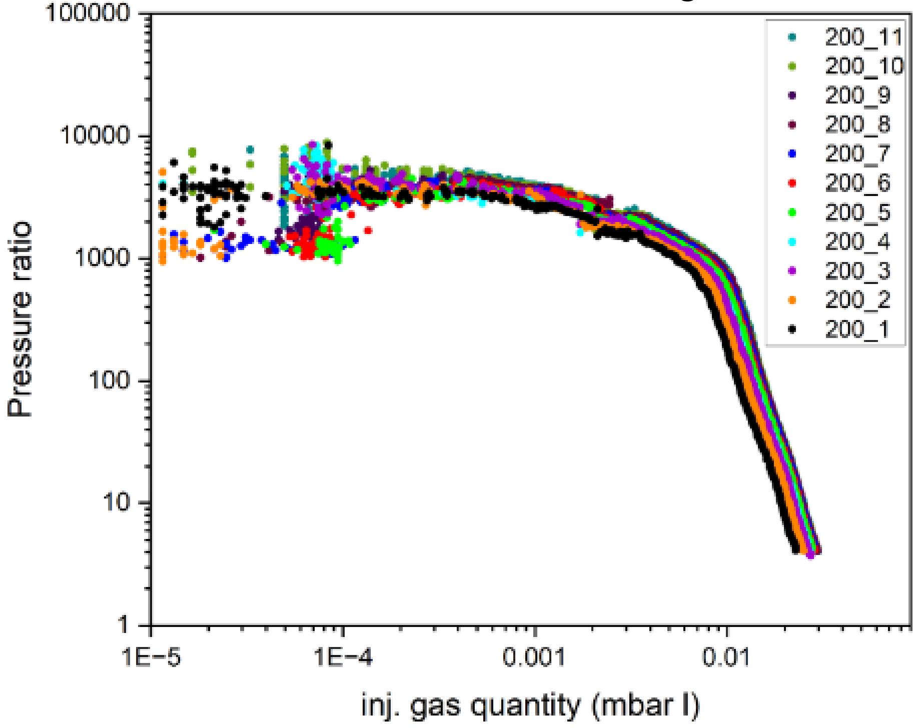


	Zr			
	Dense		Columnar	
T_{act} (°C)	C_{CO} (CO/cm^2)	C_{CO} (ML)	C_{CO} (CO/cm^2)	C_{CO} (ML)
180_1	8.70×10^{14}	1.74	6.44×10^{14}	1.29
180_2	8.29×10^{14}	1.66	6.16×10^{14}	1.23
180_3	7.55×10^{14}	1.51	5.93×10^{14}	1.19
180_4	7.06×10^{14}	1.41	5.65×10^{14}	1.13
180_5	6.63×10^{14}	1.33	5.72×10^{14}	1.15
180_6	6.13×10^{14}	1.23	5.47×10^{14}	1.10
180_7	6.11×10^{14}	1.22	5.44×10^{14}	1.09
180_9	6.21×10^{14}	1.24	5.98×10^{14}	1.20
180_10	6.04×10^{14}	1.21	5.98×10^{14}	1.20
180_11	5.63×10^{14}	1.13	4.63×10^{14}	0.93
180_12	5.83×10^{14}	1.17	5.72×10^{14}	1.14
180_13	5.65×10^{14}	1.13	6.10×10^{14}	1.22
180_14	5.93×10^{14}	1.19	5.75×10^{14}	1.15
180_15	6.46×10^{14}	1.29	5.24×10^{14}	1.05
180_16	5.65×10^{14}	1.13	5.91×10^{14}	1.18

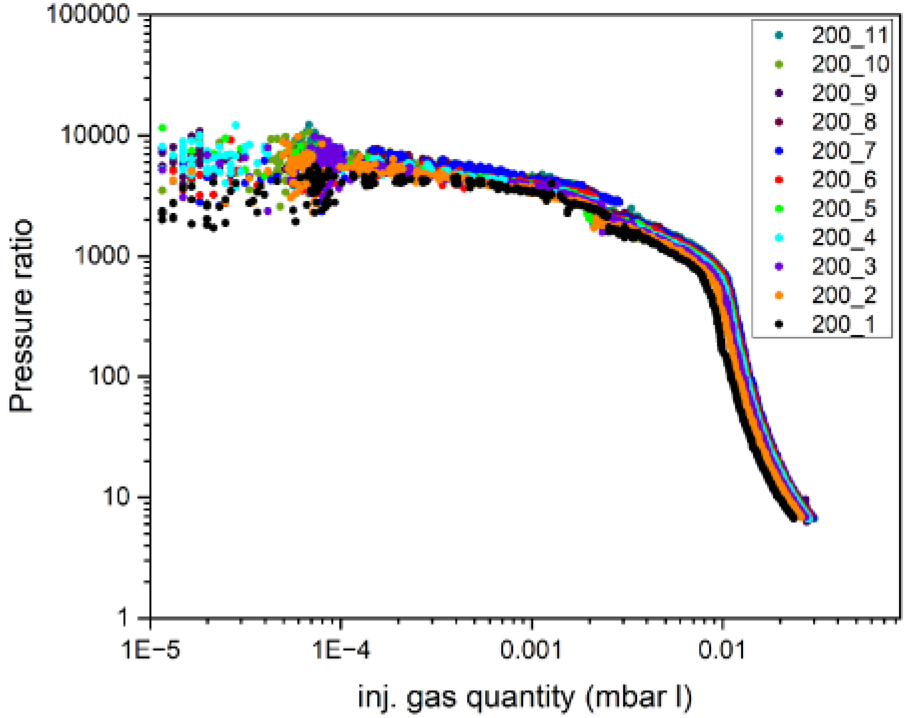
CO pumping – activations to 200 °C

- 11 activations at 200 °C degrees
- For columnar coating, until the last activation, slight increases in pressure ratio was visible
- For dense coating, until the last activation, slight increases in pressure ratio was visible

Columnar coating



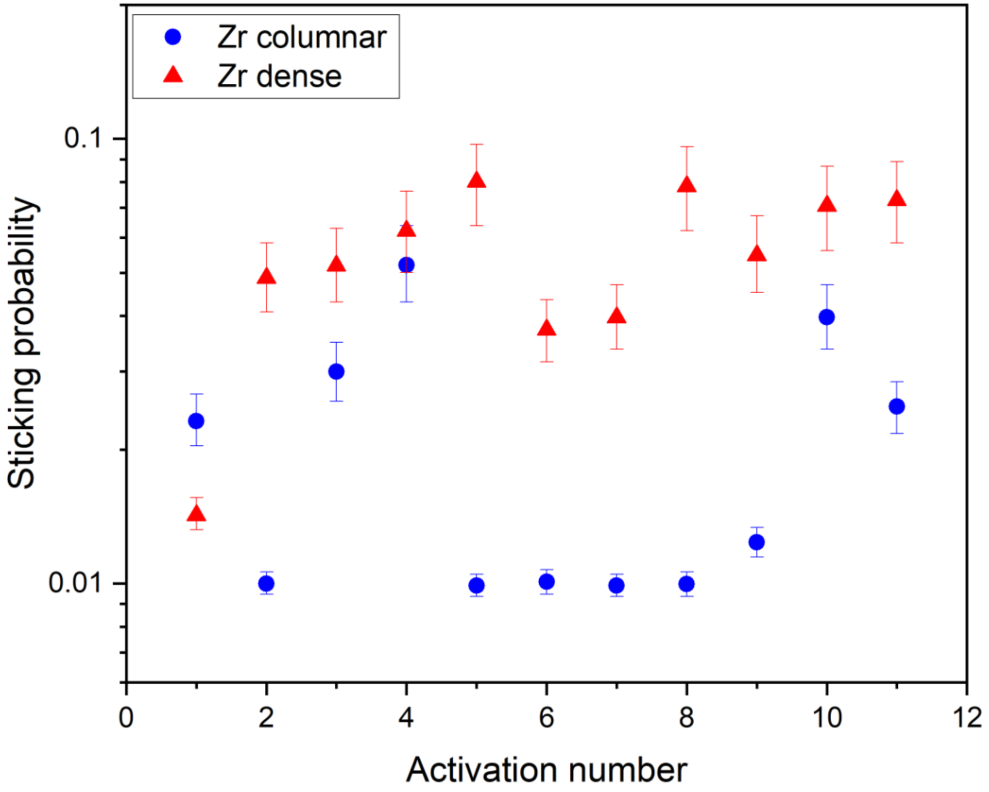
Dense coating



CO pumping – activations to 200 °C

CO sticking probability and pumping capacity

- Slow increase in capacity
- Two three-day activations done at the end



	Zr			
	Dense		Columnar	
T_{act} (°C)	C_{CO} (CO/cm^2)	C_{CO} (ML)	C_{CO} (CO/cm^2)	C_{CO} (ML)
200_1	8.97×10^{14}	1.79	8.73×10^{14}	1.75
200_2	9.87×10^{14}	1.97	9.64×10^{14}	1.93
200_3	1.04×10^{15}	2.07	1.04×10^{15}	2.08
200_4	1.08×10^{15}	2.15	1.04×10^{15}	2.09
200_5	1.06×10^{15}	2.12	1.06×10^{15}	2.13
200_6	1.10×10^{15}	2.20	1.09×10^{15}	2.19
200_7	1.13×10^{15}	2.25	1.10×10^{15}	2.20
200_8	1.13×10^{15}	2.25	1.13×10^{15}	2.26
200_9	1.14×10^{15}	2.28	1.12×10^{15}	2.24
200_10	1.10×10^{15}	2.19	1.13×10^{15}	2.26
200_11	1.15×10^{15}	2.30	1.14×10^{15}	2.28

Conclusions

Future developments

Sample preparation

- Continued dense vs columnar coating tests (both TiZrV and Zr)
  - Columnar is difficult to achieve it on narrow tubes, thickness is less uniform on columnar samples
  - Tests with more dense structures coated at lower pressures to continue
- Continued surface analysis to evaluate NEG thickness profile along the sample
  - Upgrade of the deposition system to improve the magnetic field generation
- Different OFS-Cu profile will be used in PETRA IV; tests should be repeated and conductivity of the bulk should be measured again
  - Extrusion process could impact the resistivity of the bulk copper
  - Cleaning procedure for silver-bearing copper is significantly different
- Roughness measurements of as-received, etched and coated samples (OFS-Cu profile with a cooling channel)
  - So far within limits but more tests are needed to check the roughness along the cross section

Bent chamber with a cooling channel for PETRA IV girder prototype

Future developments

Sample asessment

- Sticking probability/capacity measurements
  - More tests with dense and columnar TiZrV and Zr to continue
- NEG ageing tests
  - TiZrV (dense and columnar) samples to be installed on the setup next
- ESD measurements
  - Thermal outgassing tests with iridium and yttriated iridium wires prior
- Resistivity measurements
  - Test stand will potentially be improved to allowed for tapered connections

- Next samples to coat:
  - Bent arc chambers (bent before coating; spacers will have to be used for the deposition)
  - Undulator chamber prototypes
  - IFAST samples for PSD

Summary

- DESY has standard cleaning, deposition and test procedures to assess the various properties of NEG coating
- 5 m long undulator chamber prototype successfully coated and tested
- Resistivity measurements, while could be improved, allow for testing tubular samples with various structures/thicknesses
- Further surface analysis (SEM, EDX) and resistivity measurements are needed to collect more data and confirm the recent results
- Gained experience will be crucial for improving the future experiments in attempt of reproducible NEG coating on various sample geometries

Thank you

Acknowledgments:

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Contact

Deutsches Elektronen-
Synchrotron DESY

www.desy.de

Rūta Širvinskaitė
MVS
ruta.sirvinskaite@desy.de
+49 408998 4335