1st I.FAST Annual Meeting 2-6 May 2022

Oleg B. Malyshev (UKRI)

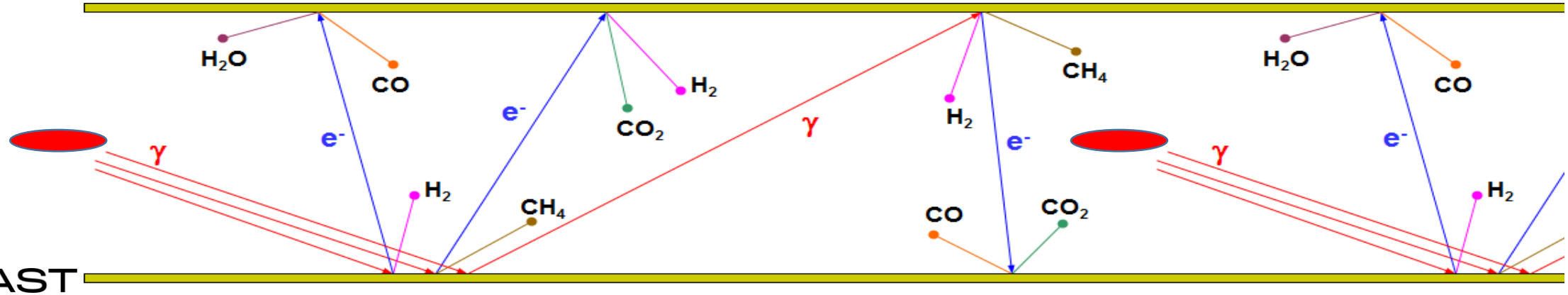
Task 10.5 leader

Vacuum in particle accelerators

All particle accelerators need vacuum. The main reason is beam-gas interaction leading to a beam quality degradation:

- Increases beam size (emittance)
- Reduces beam lifetime
- Increases radiation hazard
- Encourages recombination

- **Photon stimulated desorption (PSD)** is one of the most important sources of gas ***in the presence of synchrotron radiation (SR)*** *or any photons with E > 5-10 eV.*
- PSD can be considered as a two-step process:
- first, photons with energy >5-10 eV cause the photoelectron emission,
- then the photoelectron stimulate gas desorption.



Two concepts of the ideal vacuum chamber

Traditional:

- surface which outgasses as little as possible (‘nil’ ideally)
- surface which ***does not pump*** otherwise that surface is contaminated over time

Results in

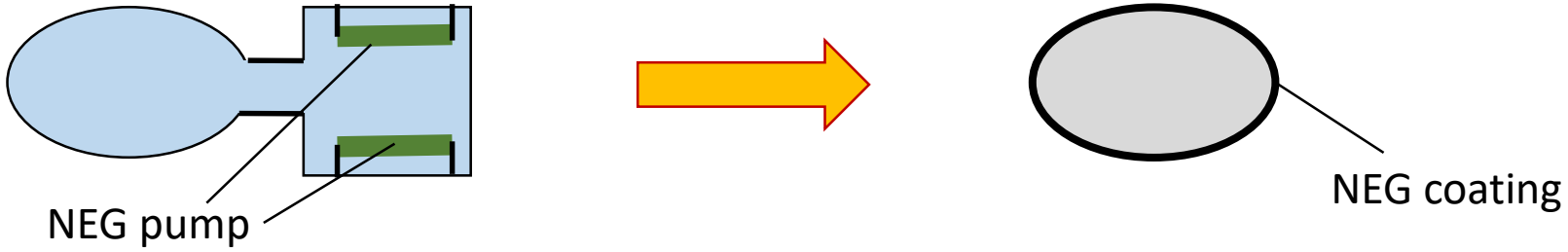
- Surface cleaning, conditioning, coatings
- Vacuum firing, *ex-situ* baking
- Baking *in-situ* to up to **300°C**
- Separate pumps

‘New’ (C. Benvenuti, CERN, ~1998):

- surface which outgasses as little as possible (‘nil’ ideally)
- a surface which ***does pump***, however, will not be contaminated due to a very low outgassing rate

Results in

- NEG coated surface
- There should be no un-coated parts
- Activating (baking) *in-situ* at **150-180°C**
- Small pumps for C_xH_y and noble gases

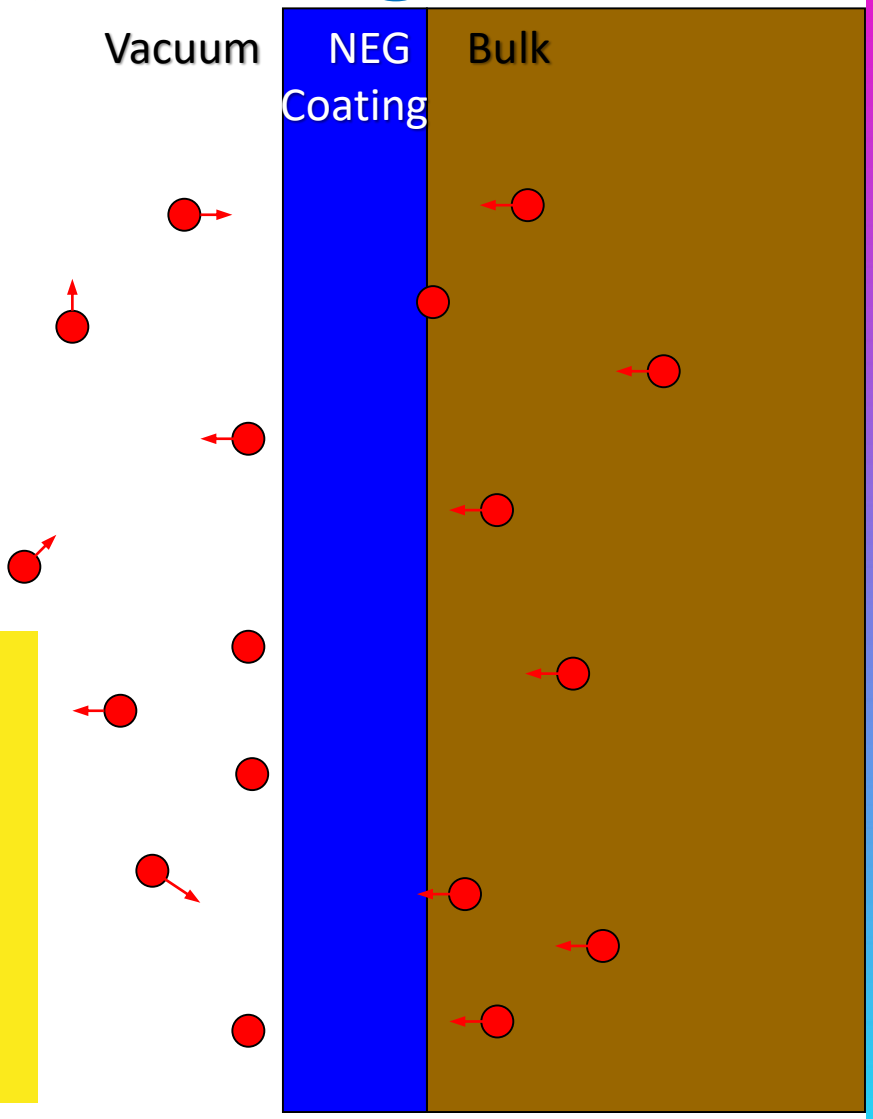


What non-evaporable getter (NEG) coating does?

1) **Reduces gas desorption:**
   - **A pure metal or metal alloy (Ti, Zr, V, Hf, etc.) film 0.5-3-μm thick without contaminants.**
   - **A barrier for molecules from the bulk of vacuum chamber.**
2) **Increases distributed pumping speed, *S*:**
   - **A sorbing surface on whole vacuum chamber surface**

providing a pumping speed of $S = \alpha \cdot A \cdot v/4$;

where α – sticking probability, A – surface area, v – mean molecular velocity



Main benefits of NEG coating:

- Can be activated at low temperature of 150-160 °C
- Meeting challenging vacuum specification at UHV or XHV
- Lower cost of vacuum system
  - ✓ Less number of pumps, thus less controllers and cables
  - ✓ Smaller size of the pumps, thus lower cost per unit
- The only solution for narrow vacuum chambers

What is really need for vacuum system design

- There are not enough PSD data for various NEG coatings.
  - A future machine vacuum deign can't be done properly without these data.
- What information have to be obtained:

- **Experimentally measured PSD yields, η, and sticking probabilities, α,** for H_2, CH_4, CO, CO_2 (*for modelling future machines*)
  - for various types of NEG coatings (composition and structure),
  - as a function of photon dose,
  - as a function of activation temperature and duration,
  - as a function of film thickness,
  - for shapes similar to vacuum chamber of future machines,
  - *etc*.

- **Practical knowledge and experience** on what happens in case of various operation issues:
  - SR induced activation, recovery rate after a vacuum accident,
  - SR induced pumping,
  - a leak during NEG activation,
  - SR beam alignment fluctuation,
  - non-uniform temperature during activation: overheated NEG, underheated NEG,
  - not uniformly coated and partially coated chambers (a chamber with an antechamber),
  - effect of storage in vacuum, in nitrogen, in argon, in air, ...,
  - NEG lifetime,
  - *other questions from machine operation experience*.

Task 10.5 objectives

- Building facilities for photon stimulated desorption (PSD) yield measurement on beamlines.
- Obtaining and analysing the photon stimulated gas desorption (PSD) experimental data from Non-Evaporable Getter (NEG) coated prototypes under conditions similar to future light sources.

	Milestone/Deliverable name	Delivery date (in months)	Content
Milestone	First NEG coated sample are installed on SR beamline at DLS and Soleil	12	Report - **Completed**
Deliverable	First PSD data from NEG coating	36	Report

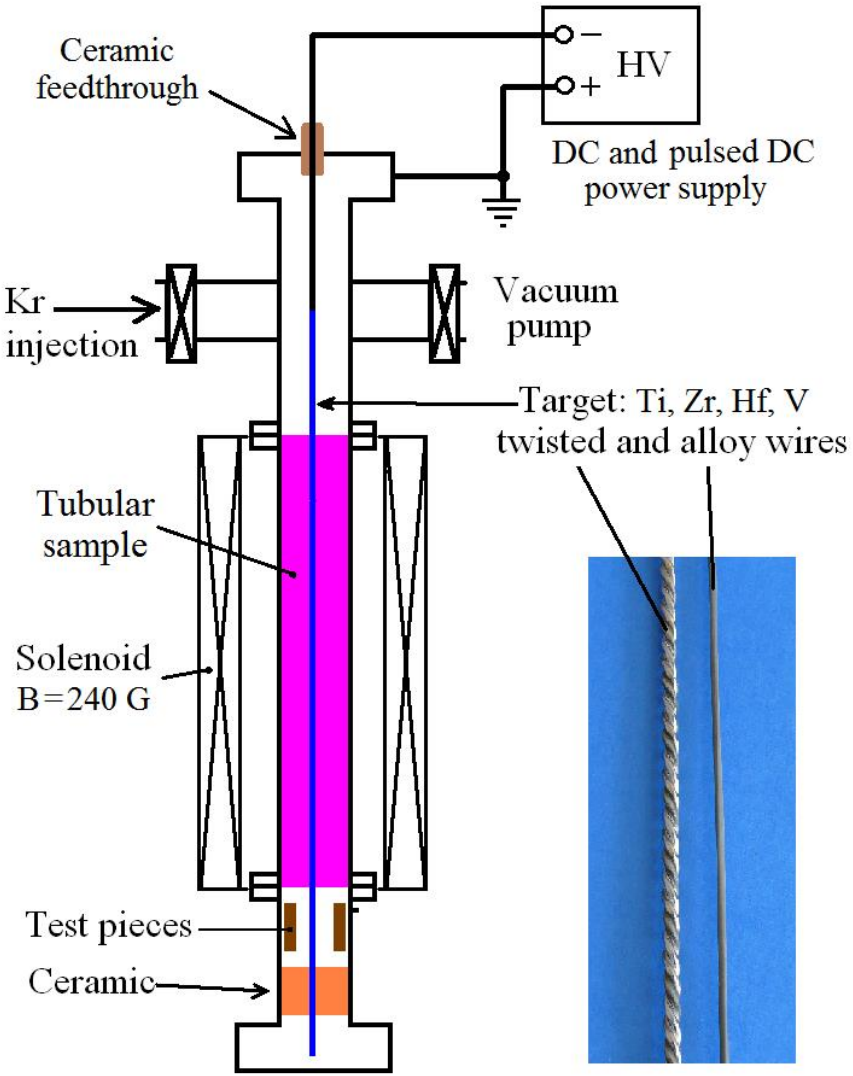
Task 10.5 partners (vacuum experts from 4 HEI)

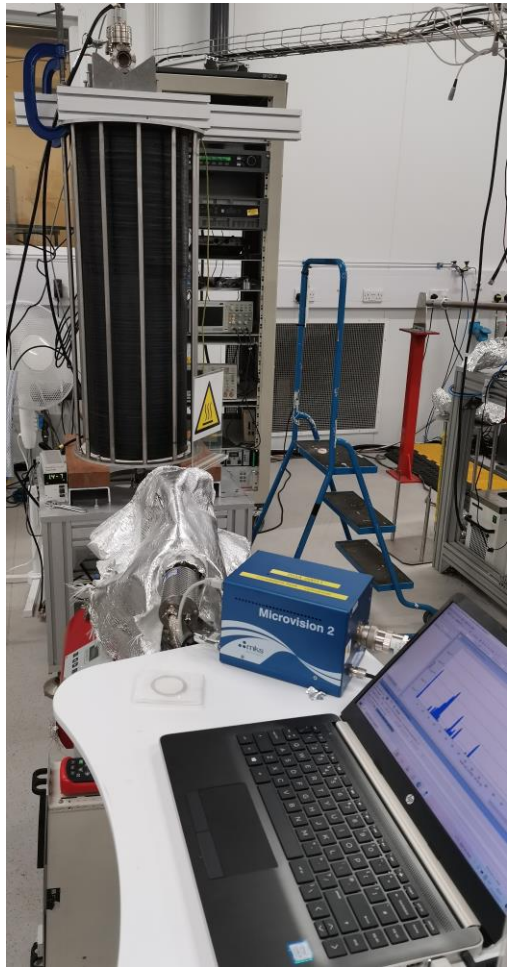
Partner	Main capability relevant to the project	Future machine
UKRI/STFC/ASTeC	• NEG deposition and characterisation • Pumping property and ESD evaluation • Gas dynamics modelling, data analysis, large vacuum system design	UK-XFEL
Diamond LS	• PSD facility at a SR beamline on Diamond • Pumping property and ESD evaluation • Large vacuum system design and operation	Diamond-2
Soleil	• PSD facility at a SR beamline on Soleil • Pumping property and ESD evaluation • Large vacuum system design and operation	Soleil-2
DESY	• NEG deposition and characterisation • Pumping property and ESD evaluation • Large vacuum system design and operation	PETRA-IV

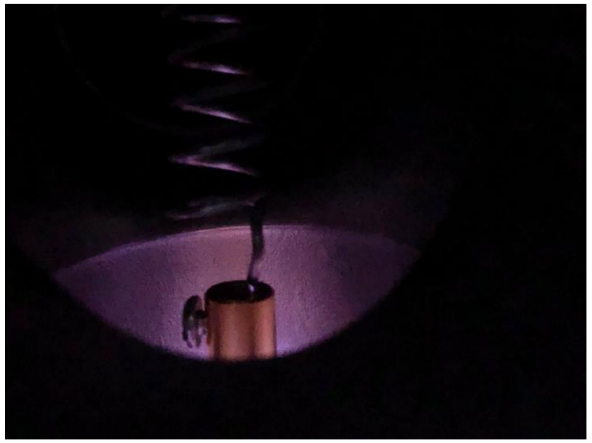
Common interest of all partners

- Vacuum chambers made of either pure copper or copper alloy
  - Surface preparation before deposition (cleaning, etching…)
  - Preparation on the deposition facility (bakeout)
- Small inner aperture (~20 mm)
- Interest to
  - New types of NEG coatings such as Zr, Ti-Zr-Hf-V
  - Alloy targets for NEG deposition
  - Low activation temperature
  - SR induced activation
  - Effect of film thickness
  - Comparison of results (on PSD, pumping properties, aging, etc.) obtained in different labs
  - Key knowledge and experience for future projects.

NEG deposition facility at UKRI (Daresbury Laboratory)





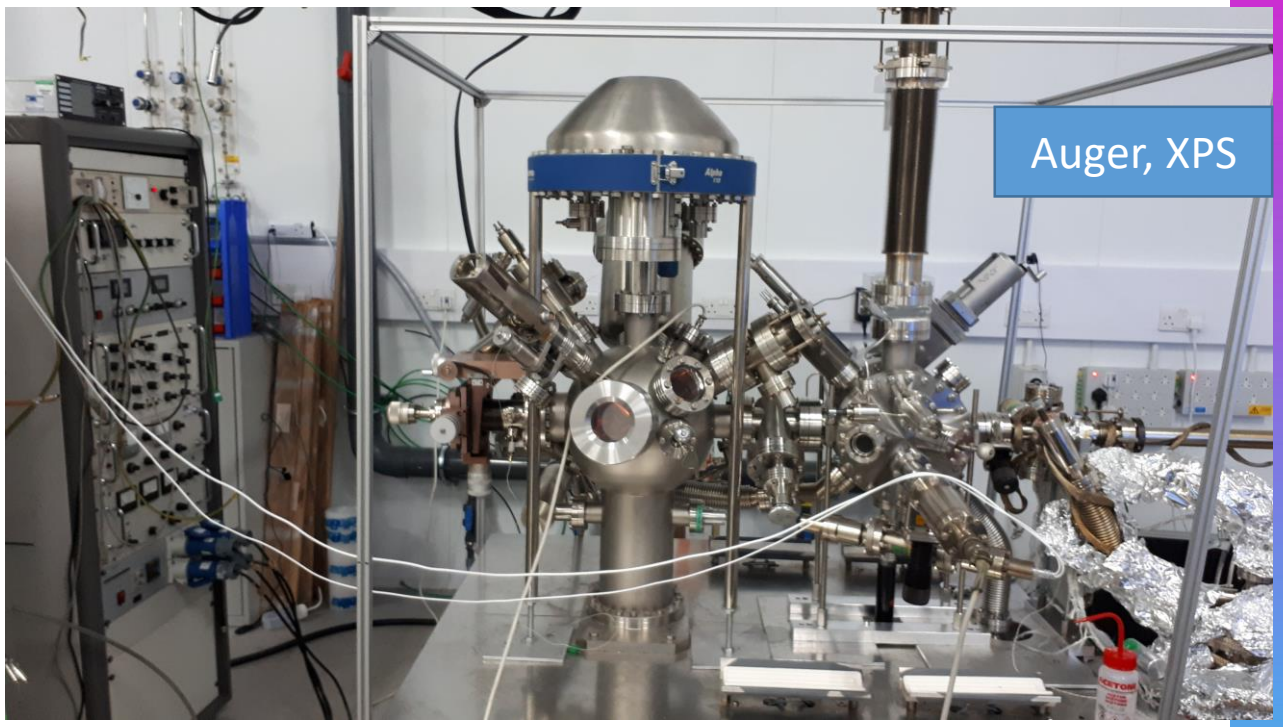


- The facility is used for a routine coating of tubes with
  - a length of 0.5 m
  - Inner diameter 5-40 mm
  - CF16-CF40 flanges
- It has been updated with a new coil to deposit thin films on
  - tubes with length of 1 m
  - Inner diameter 5-100 mm
  - CF16-CF150 flanges.

NEG Thin Film Characterisation at UKRI (DL)

FEI inspect S50 SEM

Auger



Courtesy of R. Valizadeh

More characterisation facilities are available through collaborations with

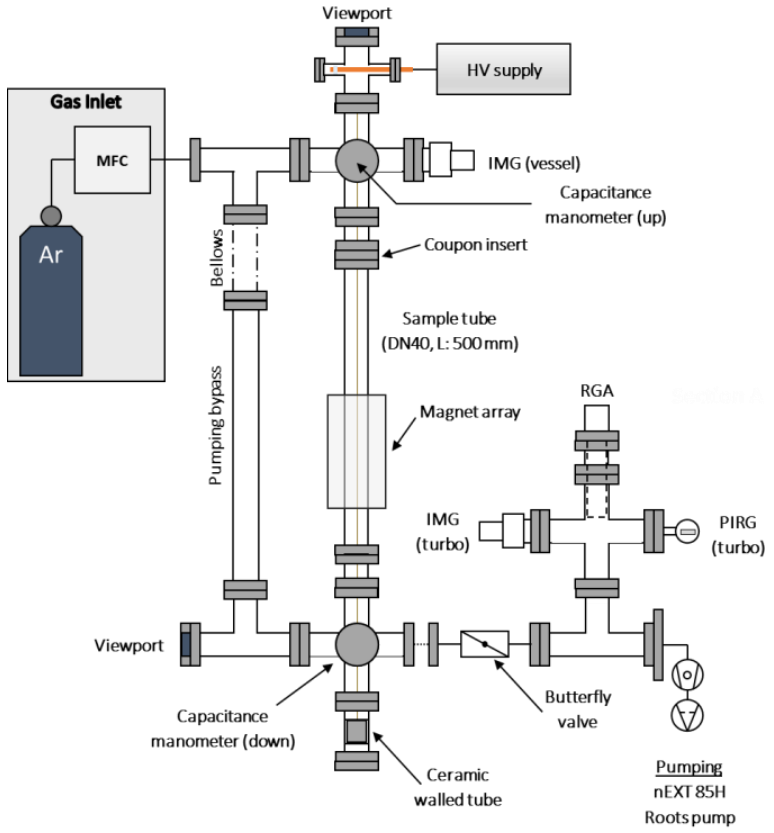
- The Materials Characterisation Laboratory at ISIS (RAL)
- CI universities
- Other UK universities
- International collaborations

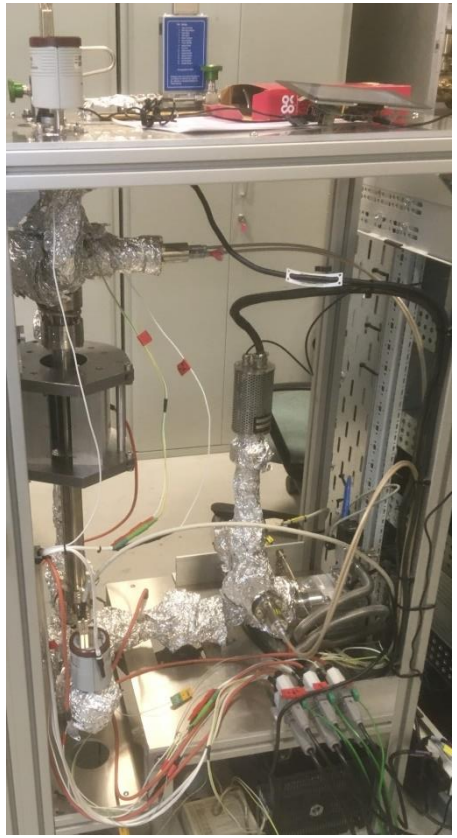
NEG deposition facility at DESY

- Support by main future project of DESY
- 1 sputtering setup for long chambers (up to 5 m)
- 1 deposition system for short chambers

Courtesy of L. Lilje and R. Sirvinskaite (DESY)

NEG deposition facility at DLS





Courtesy of C. Burrows (DSL)

Overview

- DC magnetron sputtering rig, DN40 vessels up to 0.5 m
- Base pressure around low 10^{-9} mbar range (unbaked)
- Ar sputtering gas operating between 3×10^{-3} and 1 mbar
- Moving permanent magnet (≈600 G) on rail
- Static and pulsed DC HV modes
- Twisted wire(s) tensioned by 0.5 kg mass in vacuum
- System controlled using PLC

Status:

- Increased operating pressure limit to 1 mbar
- Several short (1 hour) coating runs with Zr wire, operating at low plasma power (<4 W)

Next steps:

- Increase plasma power to approx. 15 W and perform longer coating run
- Thickness and composition determination on Si coupons

NEG deposition facility at Soleil

- There is a deposition facility at Soleil
- It could be used for a small number of samples in a future

Courtesy of Nicolas Béchu (Soleil)

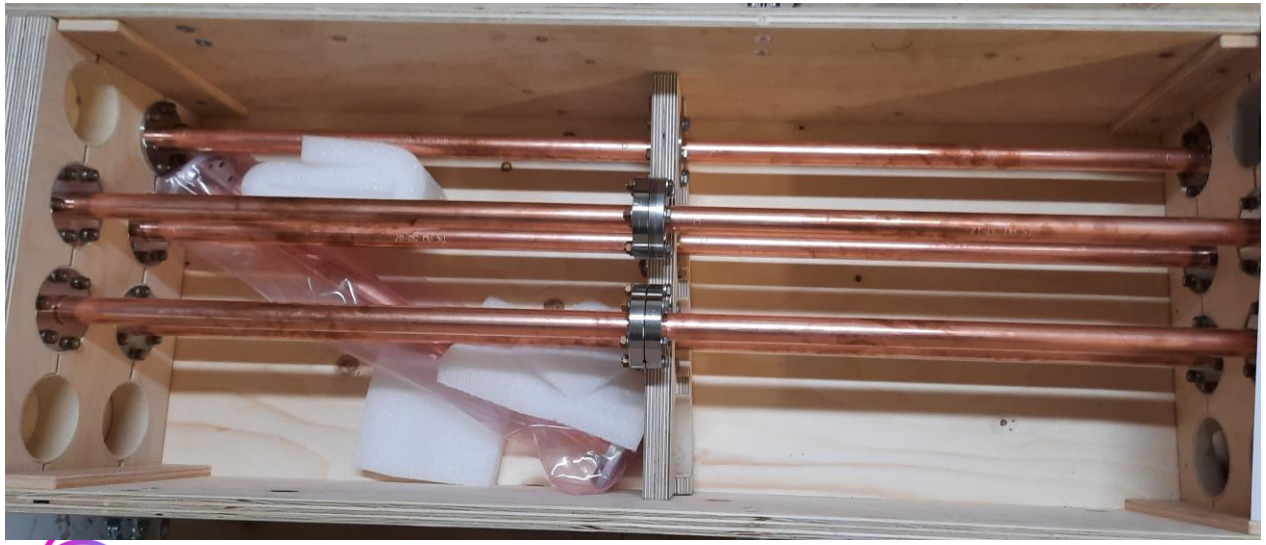
Samples for pumping properties evaluation

- It was agreed that a project *standard sample* for pumping properties evaluation is
  - made of OFHC or OFS copper samples
  - ID = 20 mm
  - L = 500 mm
  - equipped with two CF40 flanges

Courtesy of L. Lilje and R. Sirvinskaite (DESY)

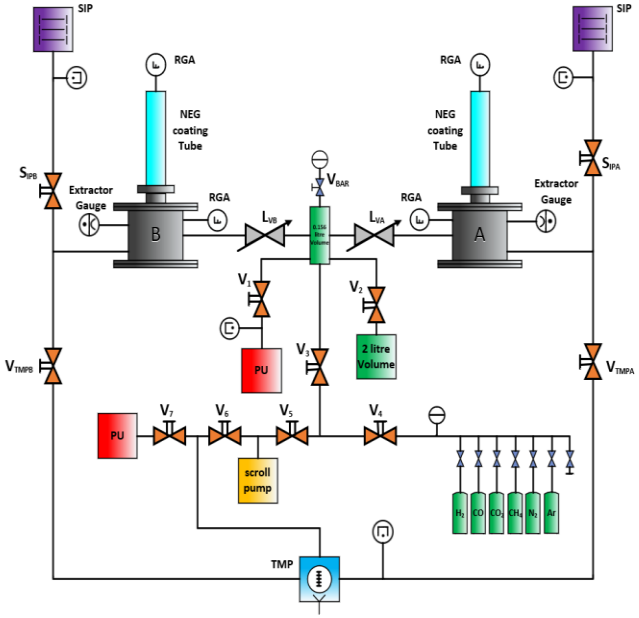
Samples for pumping properties evaluation

- 11 samples have been provided by DESY in March 2022.
  - 10 Samples have been etched with ammonium persulfate and cleaned with Elma Clean at DESY
    - following a procedure being developed in ongoing research
  - One sample have been cleaned with Elma Clean
    - but not etched with ammonium persulfate
- More samples can be produced in a future.



- 3 samples will be used for comparison of cleaning/etching procedures
  - by thermal outgassing measurements
  - by ESD measurements
- 8 samples to be coated at UKRI (DL):
  - 4 samples will be coated with Zr
  - 4 samples will be coated with Ti-Zr-V
- so ***identical samples*** will be tested in 4 labs for comparing (cross-verifying) the results obtained on different facilities

Facilities for pumping properties evaluation at UKRI (DL)

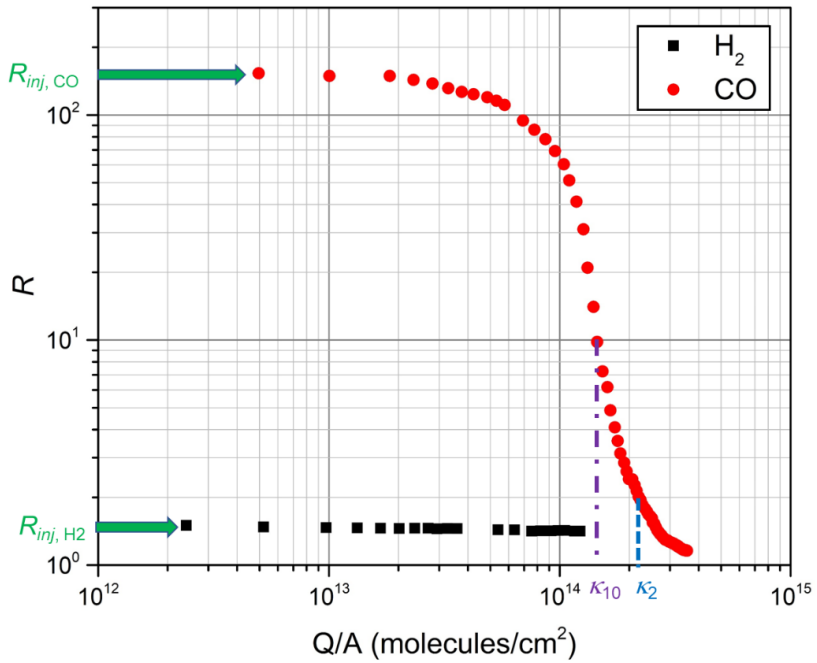


Key:

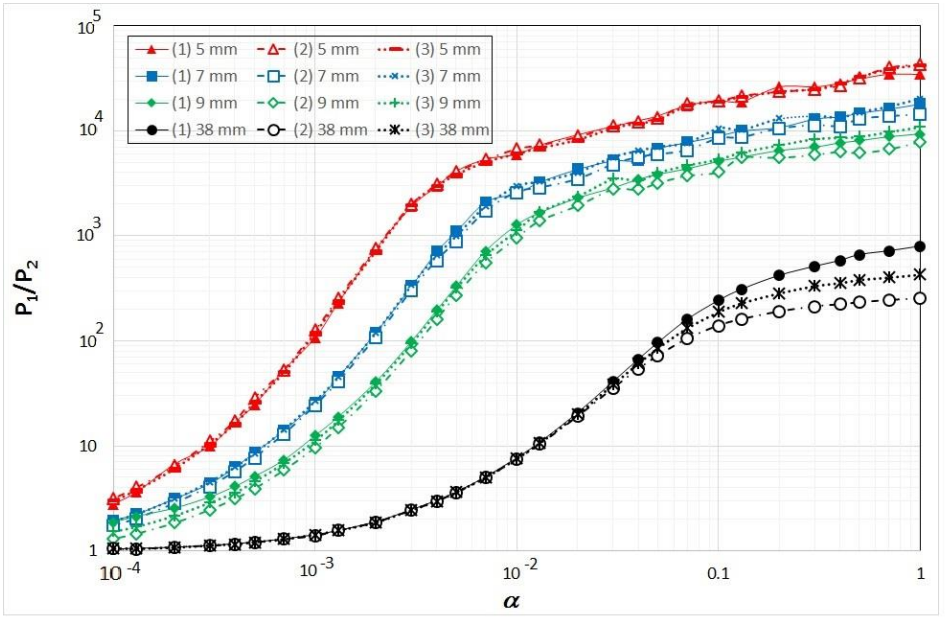
- Turbomolecular Pump (TMP)
- Sputter Ion Pump (SIP)
- Pump Unit (PU, TMP+piston pump)
- Inverted Magnetron Gauge (IMG)
- Membrane Gauge (MG)
- Residual Gas Analyser (RGA)
- Extractor Gauge (EG)
- Fine Leak Value

- The pumping properties evaluation facilities are used for a routine measurements of initial sticking probability α and sorption capacity κ.
- Up to 4 samples can be tested at the same time.

Typical result:
$R = P_{bot}/P_{top}$



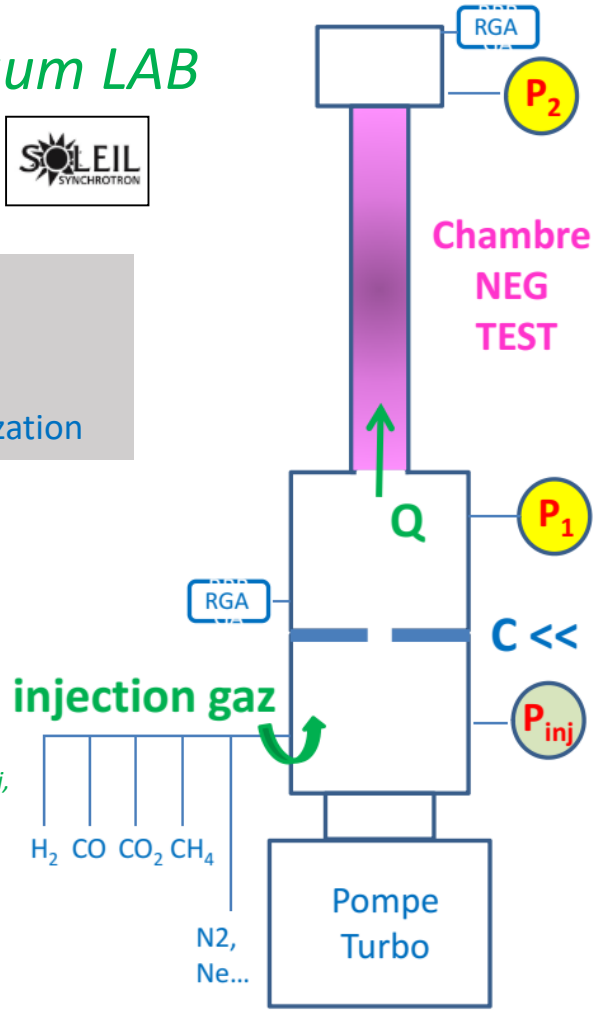
Pressure ratio $R = P_{bot}/P_{top}$ as a function of sticking probability α *obtained with TPMC modelling*



Facilities for pumping properties evaluation at Soleil

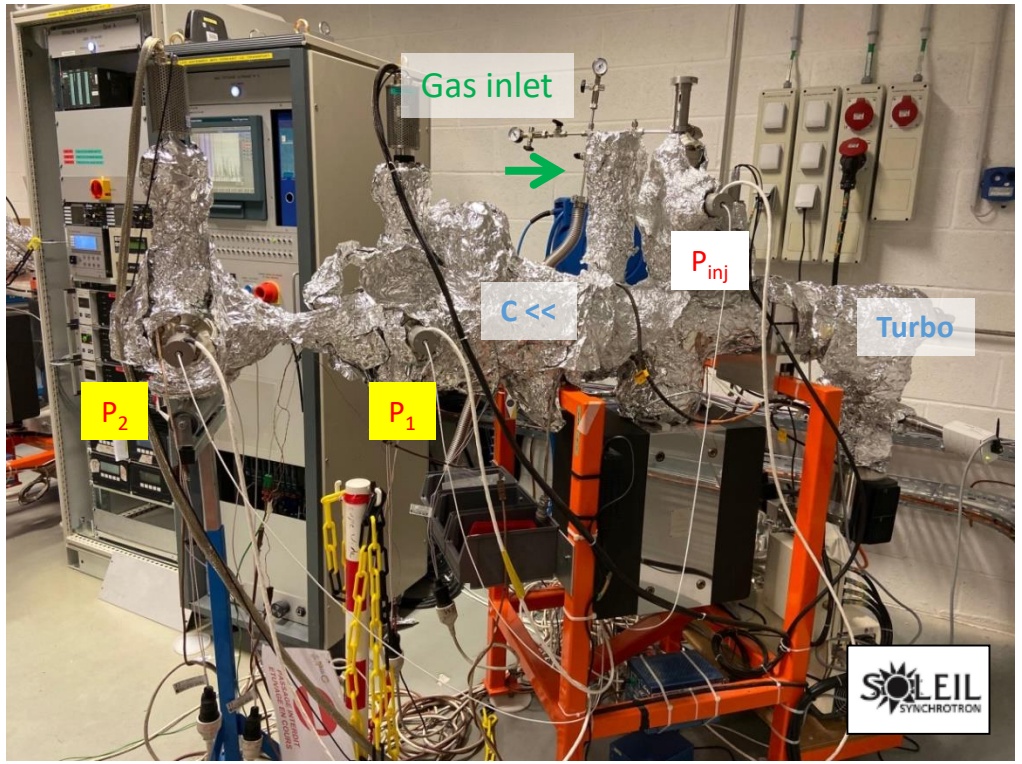
@ Vacuum LAB

- Sticking factor α
- Sorption capacity
- Activation optimization



Transmission Method

2 Transmission Method Test Benches for NEG coating characterization

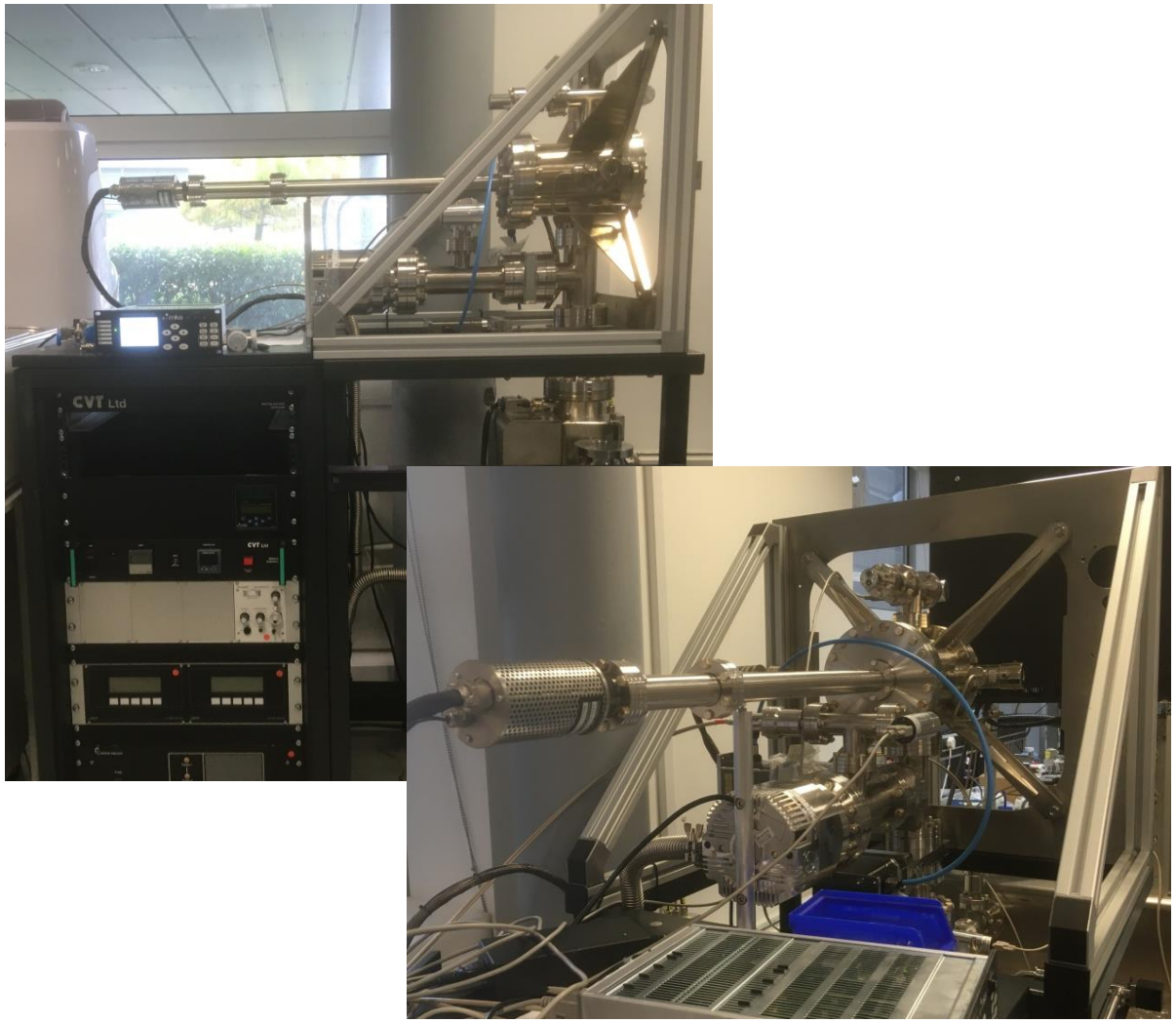


P_1/P_2 is calibrated with **MOLFLOW+** to find α

P. Costa Pinto, P. Chiggiato, A. Sapountzis, T. Sinkovits, M. Taborelli, ***CERN***
80th IUVSTA Workshop, NSRRC, Hsinchu, Taiwan (2016)

Courtesy of C. Herbeaux (Soleil)

Facility for pumping properties evaluation at DSL



Overview

- RGA-based or extractor gauge pressure ratio method to estimate sticking probability
- Pumping provided by a 300 l/s TMP and a 150 l/s SIP
- Gas injection through dedicated system based on expansion volumes
- Four gases (H_2, CO, CO_2, CH_4) let through regulated leak valve, expected operating pressures up to 10^{-5} mbar
- System to be controlled by PLC

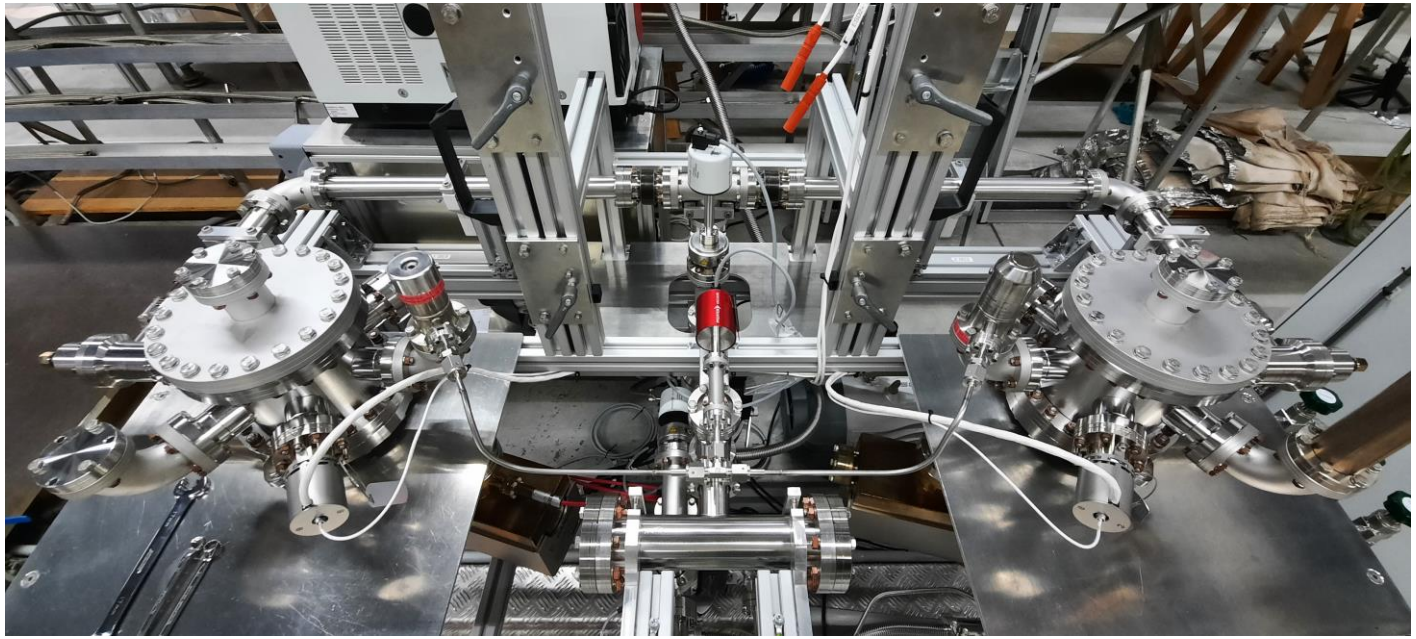
Status:

- System currently in commissioning (to be ready for samples by end of July 2022)

Courtesy of C. Burrows (DSL)

Facility for pumping properties evaluation at DESY

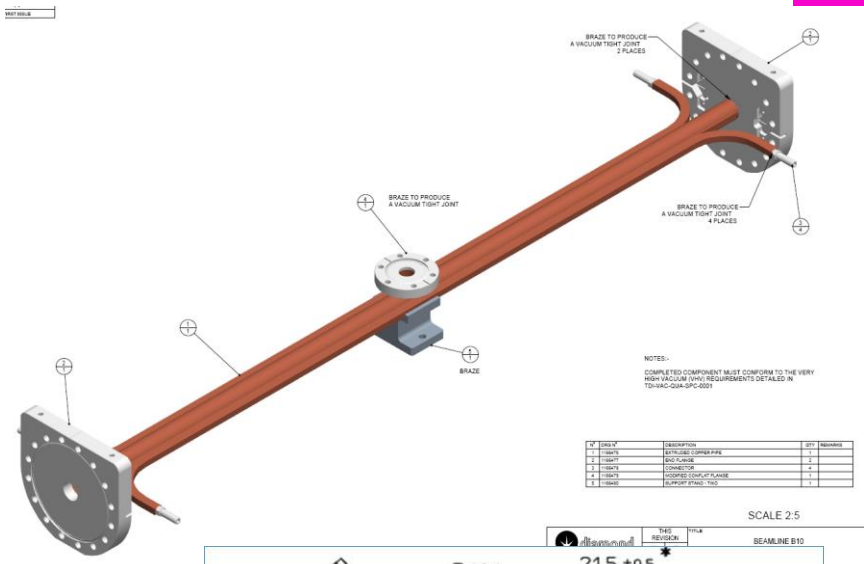
- Pumping test setup is running now after redesigning
- RGA or extractor gauge-based pressure ratio measurements
- Another mirroring system is lacking RGAs
- ESD setup (one of the two mirroring chambers) is ready for commissioning and pumping tests

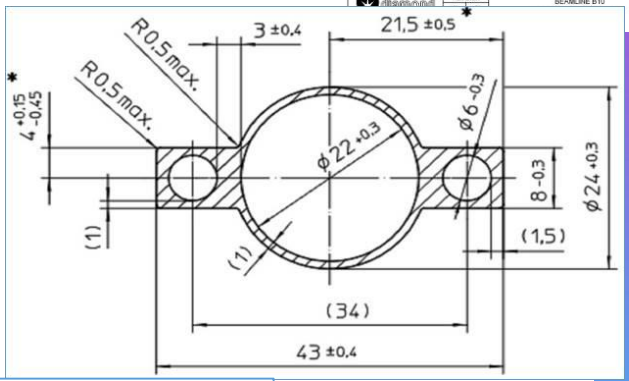


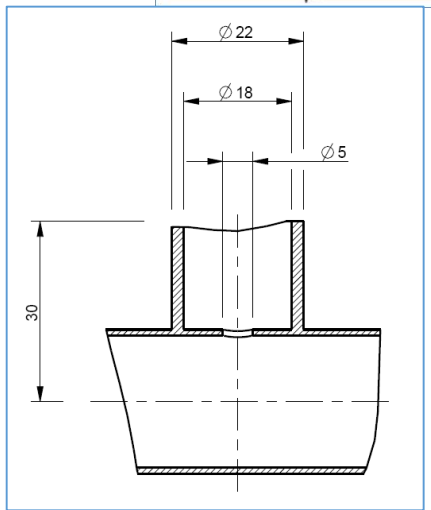
Courtesy of R. Sirvinskaite (DESY)

Sample for PSD measurements

- Samples jointly designed by Soleil and DLS
- 9 samples can be provided by Soleil by June 2022. (More samples can be produced in a future).
  - Initially, samples will be cleaned by a manufacturer
  - Later, samples will be cleaned at DESY following a procedure being developed in an ongoing research
  - Initially two samples will be coated with Zr at UKRI (DL),
    - so *identical samples* will be initially tested in DLS and Soleil for comparing (cross-verifying) the results obtained on different facilities
  - Central port is circular with ID = 18 mm and with 5-mm hole to vessel body
  - Central and end flanges are brazed



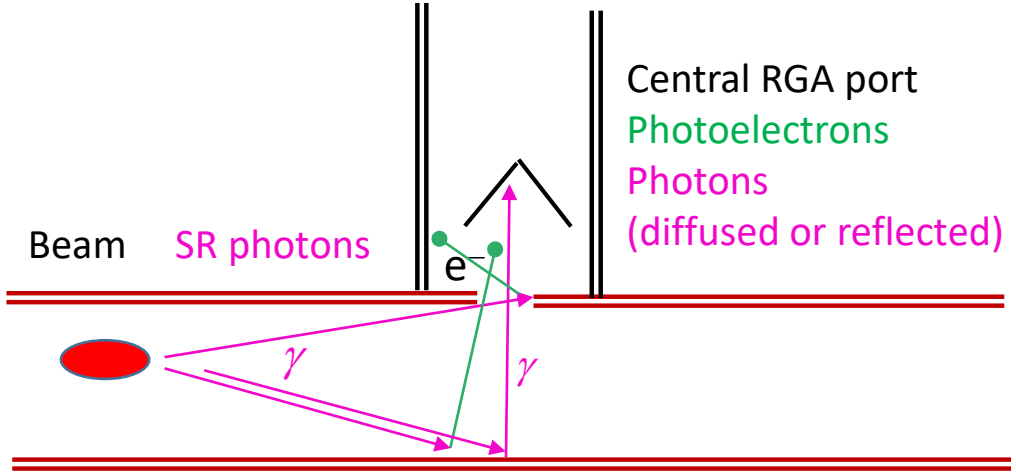


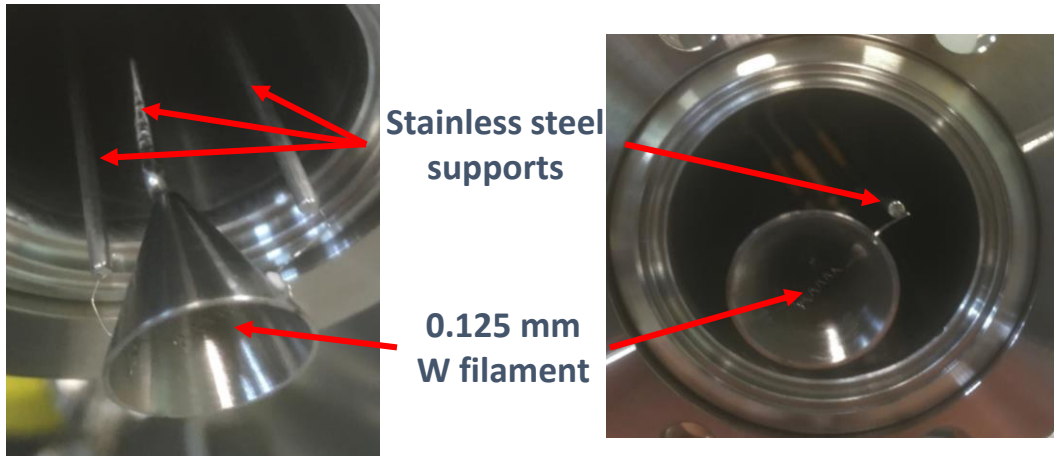


Courtesy of C. Burrows (DSL)

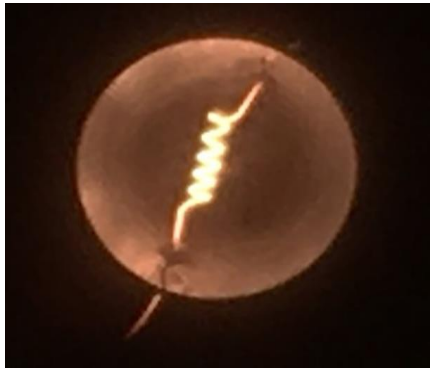
Central port

- A great attention was paid to the *central port* design as it should allow an accurate measurements of pressure inside NEG coated chamber irradiated by SR photon:
  - Low thermal outgassing
  - Low PSD and ESD inside the port
- Solution:
  - Small entrance hole
  - Covered by stainless steel cone:
    - 15 mm diameter, 0.5 mm wall thickness,
    - 3.4 mm clearance to wall
  - Filament to condition the cone with electrons
    - operated up to 3 A at ±300 V



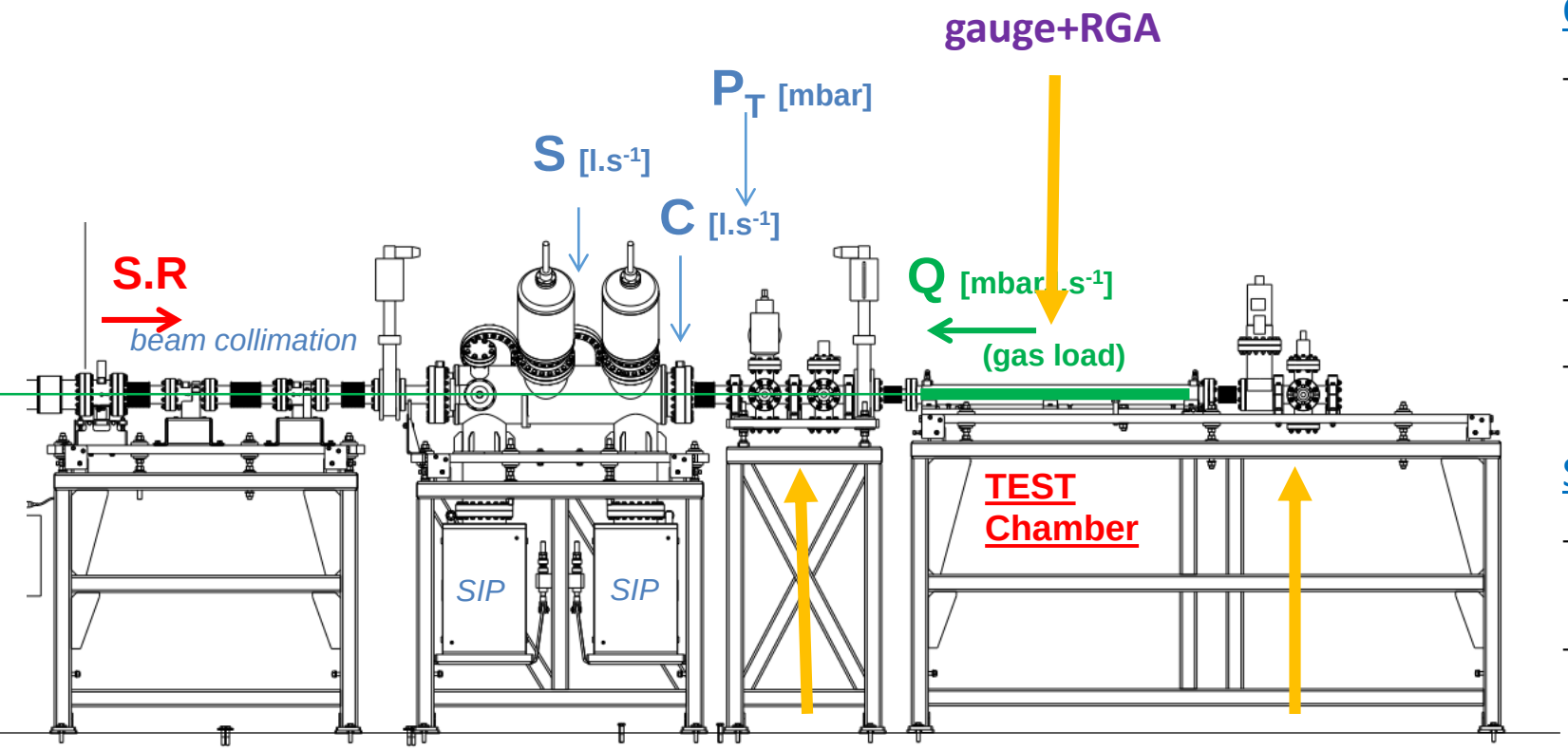


Filament glow at 1.5 A



Courtesy of C. Burrows (DSL)

An updated facility for PSD studies on SR beamline at Soleil



Courtesy of C. Herbeaux (Soleil)

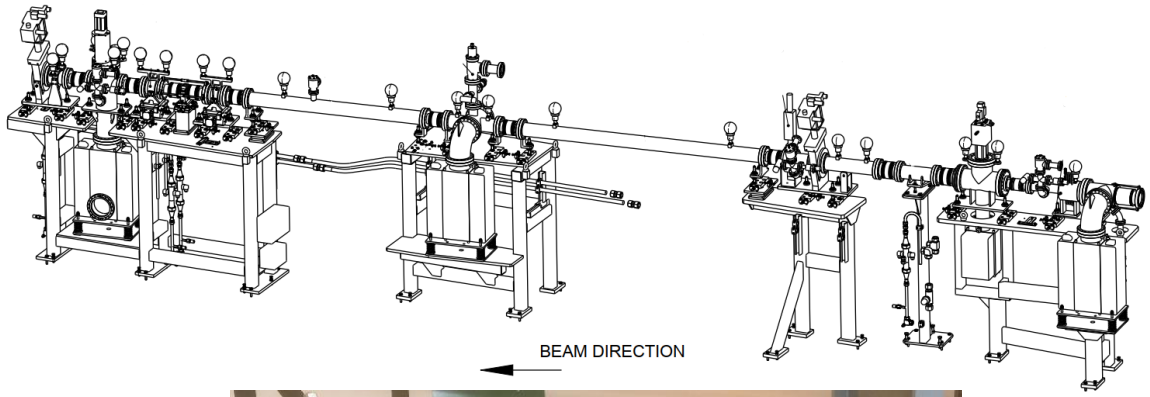
Overview

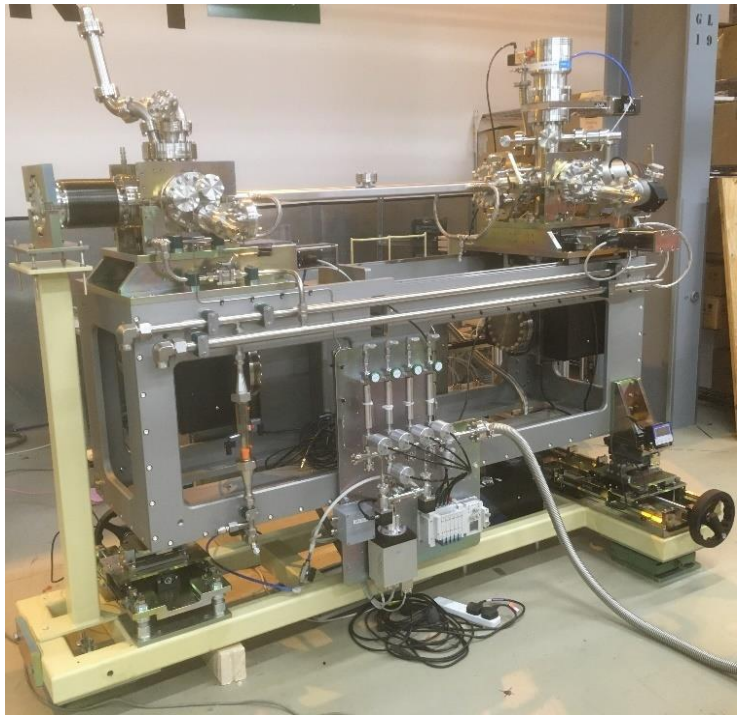
- Typical Soleil dipole front-end:
  - Photon flux: 3.4×10^{14} photon/(s·mrad·mA)
  - Critical energy: 8.379 keV
- Beam width at test piece 1-50 mm
- Test vessel angular range of up to 30 mrad (1.8°)

Status

- Front-end section: reconditioned during 2020
- Experimental end-station: currently under operation with samples coated with NEG at SAES getters
- New OFHC copper samples for SR studies designed and procured (9 pcs). Delivery expected in June 2022.
- The samples will be NEG coated at UKRI (DL) and installed during a Soleil shutdown in August 2022

A facility for PSD studies on SR beamline at DSL





Courtesy of C. Burrows (DSL)

Overview

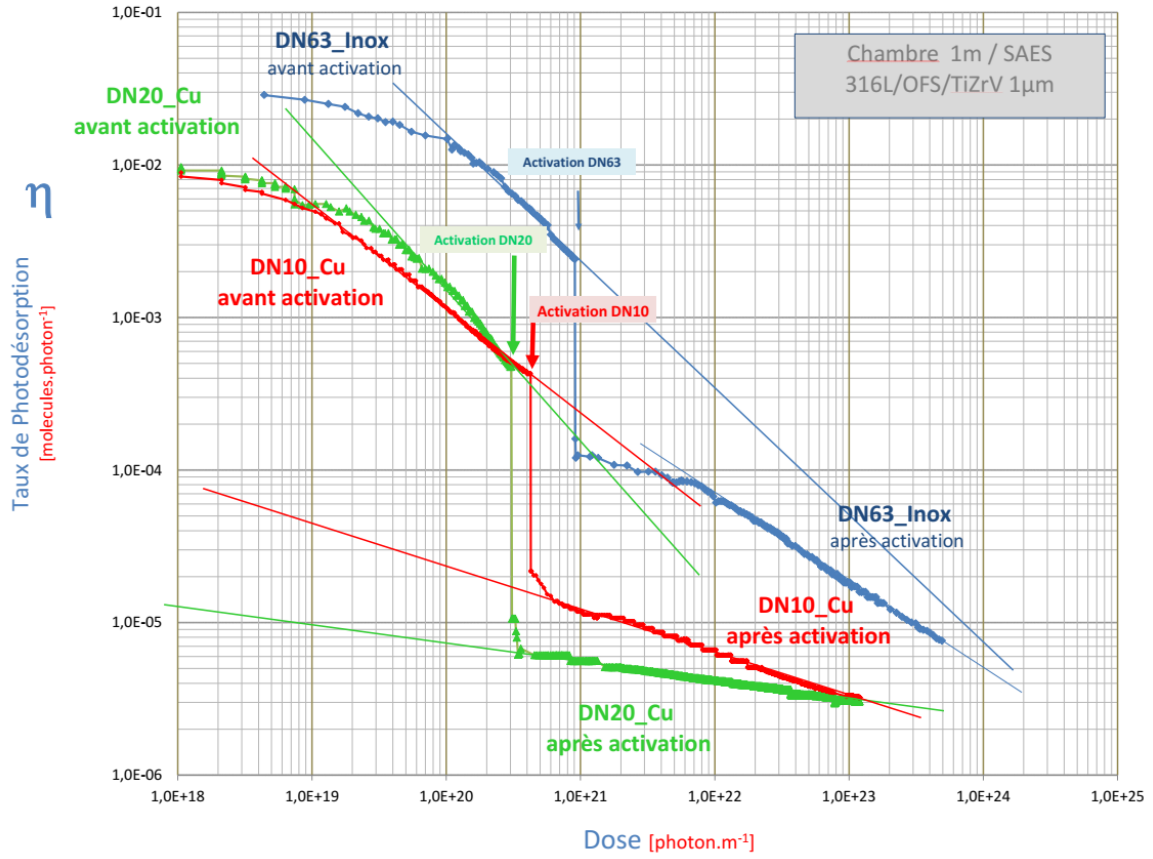
- Typical DLS dipole front-end (FE10B):
  - Photon flux: 3.86 x 10^{14} photon/(s·mrad·mA)
  - Critical energy: 8.379 keV
- Beam width at test piece up to 20 mm
- Test vessel angular range of up to 60 mrad (3.5^{o})
- Two 300 l/s SIPs and a 300 l/s TMPs for gas injection or coating saturation measurements

Status

- Front-end section: installed November 2020
- Experimental end-station: currently under post-installation conditioning with a stainless steel DN40 DLS type sample vessel (uncoated)
- Stainless steel DN40 DLS type sample NEG coated at UKRI (DL) was be installed during a DLS shutdown:
  - In March 2022
- 1st copper sample coated with NEG could be installed during a following DLS shutdown
  - In August 2022

Results of PSD testing at Soleil

- Three 1-m long, water cooled vacuum chambers with ID = 63, 20 mm and 10 mm were installed on the PSD beamline.
- The chambers were previously NEG coated at SAES with a 1-µm thick TiZrV film.
- Total PSD yields calculated for initial SR irradiation before NEG activation, followed by activation to 200 °C for 24 h and continuing with SR irradiation.



Task 10.5 coordination activities

- Task 10.5 has started with two preparatory meetings:
  - on 2nd March and 7th April 2021
- Task 10.5 meeting take place every month:
  - 1st Task 10.5 (kick-off) meeting on 12th May 2021
  - 2nd Task 10.5 meeting on 15th Sep 2021
  - 3rd Task 10.5 meeting is scheduled on 12th May 2022
  - Monthly briefing Task 10.5 meeting
- Deposition and testing facilities are operational at UKRI and DESY
- SR beamlines have been design, built and are already in operation at DLS and Soleil
- Milestone Report MS47 has been submitted on 1/4/2022

Summary

- Task 10.5 team works in full capacity according its plan
- All necessary capabilities exist at least with two partners
  - Deposition facilities are operational at UKRI and DESY, in conditioning at DSL, can be used at Soleil
  - Pumping property evaluation facilities are operational at UKRI, DESY and Soleil, in conditioning at DSL.
- SR beamlines
  - Reconditioned and is in operation in Soleil
    - tested with a sample coated with NEG at SAES Getters
  - Have been design, built and are already in operation at DLS
    - A stainless steel sample coated with NEG at UKRI has been installed for PSD measurements
- Samples:
  - 11 samples for pumping property measurements have been produced and delivered to UKRI:
    - 8 samples are ready for NEG coating and testing in 4 labs
    - 3 samples are used for comparing copper cleaning procedures by TD and ESD measurements
  - 9 samples for PSD measurement have been designed and are under production
    - to be delivered for NEG coating in June and
    - to be installed on SR beamlines in August 2022
- Milestone MS48 report submitted on time

Acknowledgment (Task 10.5 team)

DLS

- Matthew Cox
- Chris Burrows
- Hugo Shiers

DESY

- Lutz Lilje
- Ruta Sirvinskaite
- Nils Plambeck
- Ralph Böspflug
- Sven Lederer

Soleil

- Christian Herbeaux
- Nicolas Béchu
- Vincent Le Roux

UKRI (STFC/DL/ASTeC)

- Oleg Malyshev
- Reza Valizadeh
- Eleni Marshall
- Adrian Hannah
- James Conlon

iFAST

Thanks for your attention

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