I.FAST

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MILESTONE REPORT

First NEG coated samples are installed on SR beamline at DLS and Soleil

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ABSTRACT

Report is related to achieving I.FAST Milestone 48 (First NEG coated samples are installed on SR beamline at DLS and Soleil). A new dedicated SR beamlines was built and tested at DLS, a sample coated with NEG at UKRI/STFC has been installed and ready for a test immediately after restarting DLS operation (after scheduled shutdown) from mid-April. An existing dedicated SR beamlines at Soleil Synchrotron was recommissioned and tested with 3 samples coated with NEG at SAES Getters. Thus, the I.FAST Milestone 48 (First NEG coated samples are installed on SR beamline at DLS and Soleil) has been met.

For more information on IFAST, its partners and contributors please see https://ifast-project.eu/

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Executive summary

This Report is related to achieving I.FAST Milestone 48 (First NEG coated samples are installed on SR beamline at DLS and Soleil). A new dedicated SR beamlines was built and tested at DLS, a sample coated with NEG at UKRI/STFC has been installed and ready for a test immediately after restarting DLS operation (after scheduled shutdown) from mid-April. An existing dedicated SR beamlines at Soleil Synchrotron was recommissioned and tested with 3 samples coated with NEG at SAES Getters. Thus, the I.FAST Milestone 48 (First NEG coated samples are installed on SR beamline at DLS and Soleil) has been met.

1 Introduction

Non-evaporable getter (NEG) coatings, developed at CERN in 1990s [1-4], are already widely used in particle accelerator vacuum chambers to meet challenging UHV/XHV specifications [5-15]. An internal surface of a tubular vacuum chamber is usually coated from a target comprising the material(s) required for deposition, it could be either a single metal Zr rod [16], or formed from three twisted wires made of different metals such as Ti, Zr and V [4,17] or an alloy rod of these metals [18-20]. After installation, such a NEG-coated chamber should be activated by baking to 180 °C (or higher) for 24 hours. A large distributed pumping speed allows meeting the vacuum specification with fewer pumps of much smaller size.

For designing vacuum systems of particle accelerators (especially of a new generation of light sources) based on NEG coating, one need an experimental data of photon stimulated desorption and sticking probability of H_2, CH_4, CO and CO_2 for NEG coating at different conditions [21]:

- As a function of accumulated photon dose,
- After different activation temperatures,
- After saturations with sorbing gases and after exposures to air,
- For different composition and morphology of NEG film,
- SR induced re-activation (without heating)
- etc.

Photodesorption experiments with NEG coated chambers were performed in past [22,23]. However, these results are limited to a single sample and does not represent a variety of NEG coatings developed since then.

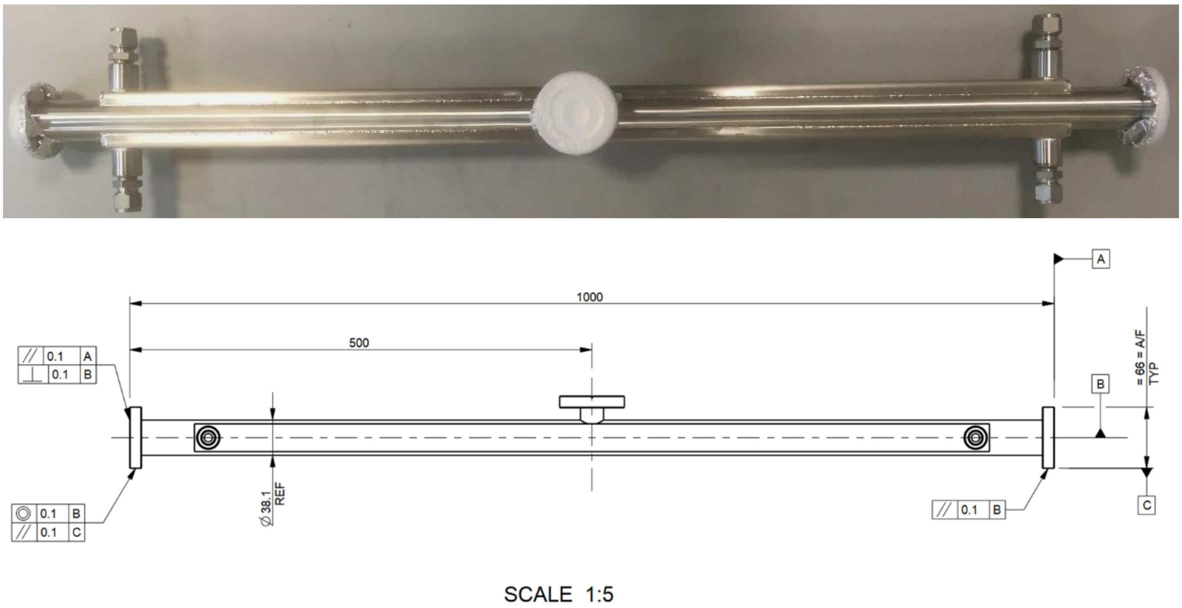
The aim of this proposal is to test a few types of new NEG coating (developed in UKRI/STFC) under synchrotron radiation in different operation scenarios. This Report is demonstrating achieving a key Milestone: installing first NEG coated samples at DLS and Soleil SR beamlines.

2 Sample design and manufacturing

2.1 Sample for 1st experiment at DLS

The base for the sample design was the so-called three-RGA method as described in Ref. [23]. That method requires an in-built port for the RGA assembly in the middle of a sample. Figure 1(a) shows a picture and Fig. 1(b) a picture of the sample designed and produced at DLS for a first test for NEG coated chamber at DLS beamline.

(a)

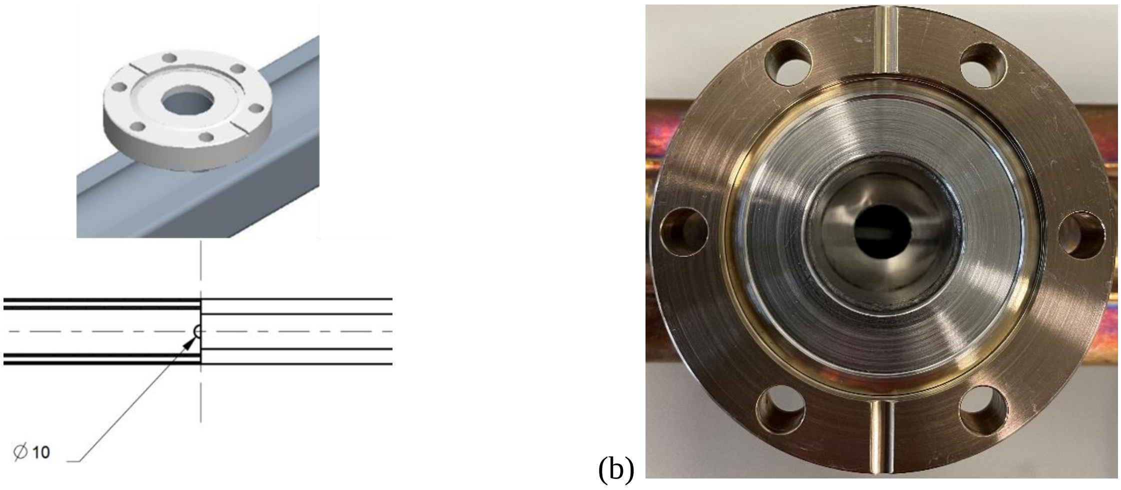


(b)

Figure 1: (a) Photograph of the first sample for NEG coating and testing under SR at DLS beamline, (b) A dimensioned drawing of the same test piece.

The first DLS NEG-coated chamber consists of a 1-m long 304L stainless-steel vessel with an inner diameter of 34.9 mm. Water cooling channels are welded either side of the chamber to manage the heat load from the photon exposure during measurements, the use of two channels enables both sides of the vessel to be used as needed. As mentioned above, a notable feature of this test chamber is the central port required for the three-RGA method. This consists of a short 26 mm section with an internal diameter of 21.8 mm attached to a standard DN40 flange, connecting this volume to the main body is a 10-mm hole. A representative model and diagram of this is shown in Fig. 2(a), and a picture of the physical assembly is included in Fig. 2(b).

(a)



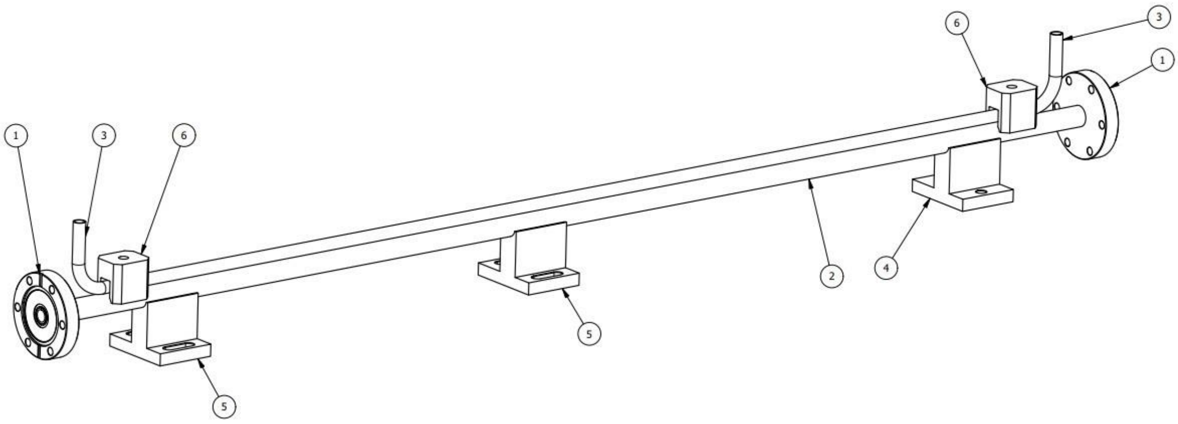
(b)

Figure 2: (a) a schematic view of the central port, (b) a photograph of the central port showing the 10 mm hole to the chamber body.

After manufacturing the sample was delivered to UKRI/STFC Daresbury Laboratory for NEG coating.

2.2 SAMPLE FOR 1ST EXPERIMENT AT SOLEIL

The first NEG coated samples were designed and manufactured with different inner diameters (ID63, ID40, ID20, ID10 mm) and different raw materials (Copper and Stainless steel), NEG coated at SOLEIL facilities or by a commercial suppliers (SAES), and irradiated on SOLEIL PSD beamline so-called *D08-1 PSD*. The samples were designed as simple water-cooled pipe of 104 centimeters long, with alignment references and stands. They were used for commissioning the SOLEIL PSD beamline, and to prove the concepts of the decrease of secondary photo-desorption yield with NEG activation and irradiated photon dose. These experiments were conducted with the classic two-pressure transmission method. The beamline will be now adapted for three-gage method in order to test samples from the IFAST partners. A schematic view of the sample is shown in Fig. 3(a), and a picture of the beam line is shown in Fig. 3(b).



(a)

(b)

Figure 3: (a) A schematic view of one of the first NEG coated samples (ID 10 mm) for testing under SR at SOLEIL PSD beamline. [1] CF40 flanges – [2] main pipe – [3] water-cooling channel – [4] fixed stands – [5] sliding stands – [6] alignment references. (b) An actual picture of the SOLEIL PSD beamline as testing under SR the first NEG coated samples (inner DN63mm SS).

2.3 DESIGN OF SAMPLES FOR FUTURE EXPERIMENTS

The main purpose of this task is to test the prototypes of vacuum chambers for future upgrades of the light sources at DLS, DESY and SOLEIL. The agreed parameters for these prototypes, so-called *PSD samples*, were inner diameter *ID* = 20 mm, length *L* = 1 m, vacuum chamber material – OFHC copper. Thus, samples should also have a middle port.

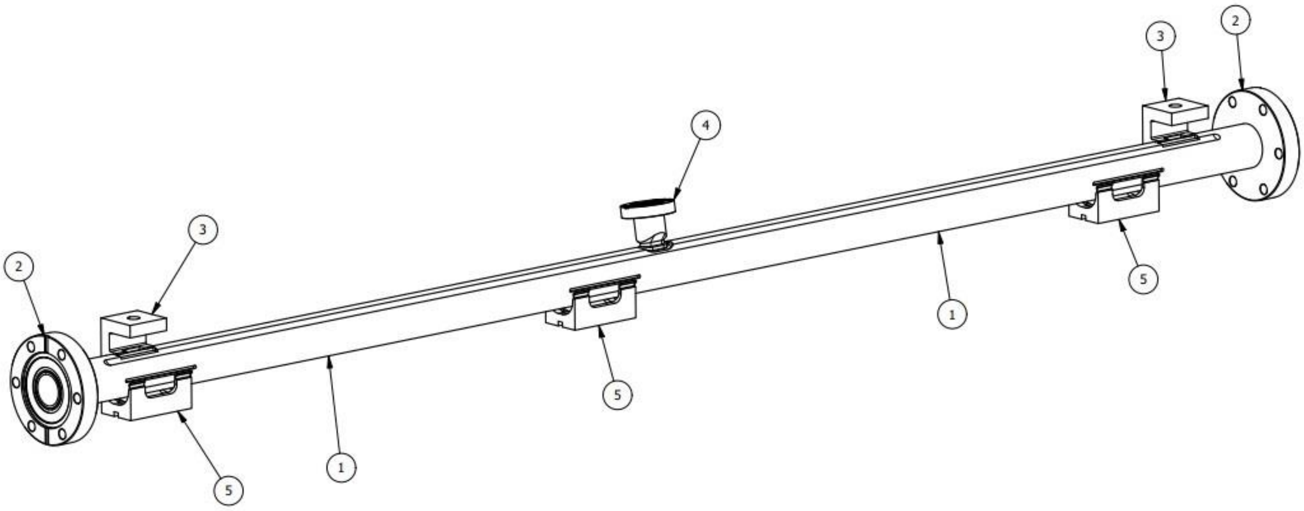


Figure 4: A schematic view of PSD Sample for NEG coating and testing under SR at DLS, DESY and SOLEIL beamlines. [1] Main DN20 Cu pipe - [2] CF40 flanges – [3] alignment references - [4] central CF16 flange for 3-gages method - [5] stands with removable feet (removed) - [not shown] above water-cooling channel.

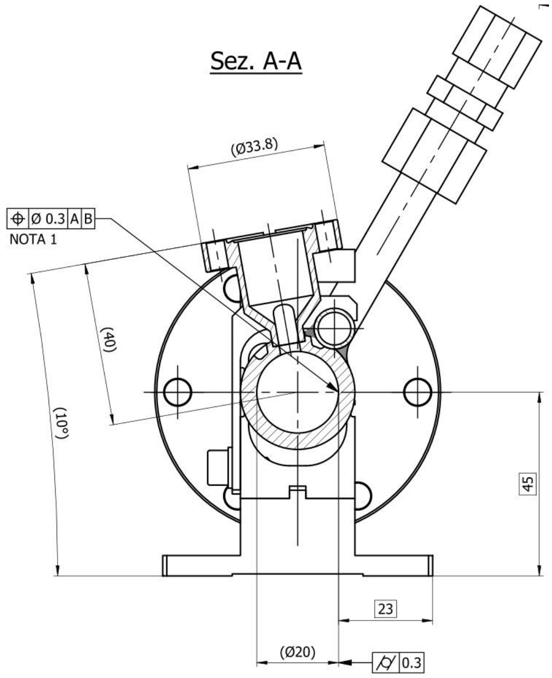


Figure 5: A schematic view of the central port cut for 3-gage measurement method.

Figure 4 shows the sample design. The design was derived from SOLEIL's and DLS's samples and adapted to I.FATS Task 10.5 partner's needs: shorten to 1 m long, removable feet, and a central port for applying the 3-gauges (or 3-RGA) foreseen methods for I.FAST Task 10.5 experiments. Figure 5 shows the design of the central flange.

At this time 9 samples have been ordered to Italian company SAES RIAL, and should be delivered ready for NEG coatings: 6 samples will be coated at STFC and 3 samples will be coated at DESY. Different coatings will be realised as twin chambers and tested at both SOLEIL and DSL beamlines. Results will be compared and analysed with all IFAST Task 10.5 partners.

3 NEG coating process

3.1 A NEW DEPOSITION FACILITY AT UKRI FOR OF 1-M LONG SAMPLE COATING WITH NEG

The NEG coating deposition UKRI/STFC Daresbury Laboratory is comprised of a 1-m long coil with a core opening of 220 mm diameter as shown in Fig. 6. The coil is air cooled while is in operation. The 1-m long DLS sample described above make an integrated part of the deposition vacuum system. Two 3-mm diametre Ti-Zr-V rods were inserted from each end of the tube which are connected to the substrate tube via two and ceramic breaks and auxiliary T parts accommodating window flanges to observe the plasma condition as well as pumping port. The deposition vacuum vessel is separated from the vacuum pump assembly by right angle whole-metal valve. This allows the deposition vessel to be initially pumped and baked ex-situ. After bakeout to 150 °C for 24 hour, a base pressure of 2×10^{-10} mbar is reached with H_2 being the dominant species in the residual gas in the vacuum. The vessel was then removed from baking chart and transferred under vacuum and secured in the centre of the 1-m long coil. The vessel was then connected to deposition vacuum chart and the connecting section was pumped for a further 12 hours before the deposition vessel is opened up to the vacuum chart.

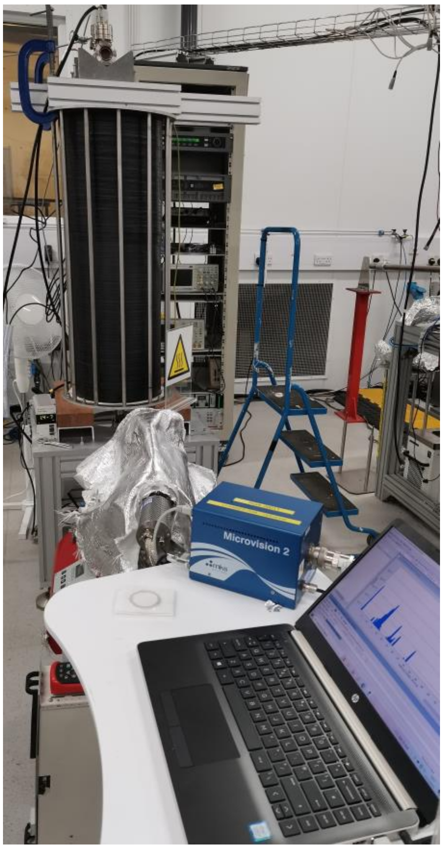


Figure 6: The 1-m NEG deposition vacuum vessel and the magnetic coil prepared for depositing NEG film.

Table 1: Deposition parameter for NEG deposition.

Power	75 W
Current	0.51 A
Voltage	146 V
Pulse frequency	350 kHz
Duty cycle	1.1 µs
Deposition time	5 hours
Deposition pressure	6×10^{-2} mbar
Mag current	23.80 A
Deposition temperature	90 – 110 °C

The deposition carried out using Advance Energy Pinnacle plus in pulsed DC mode to provide higher concurrent ion bombardment during deposition to produce a thin film in a high density structure. Krypton was used for deposition gas. Figure 7 depicts the plasma condition during the deposition taken at the bottom (Fig. 4(a)) and at the top (Fig. 4(b)) viewing port.

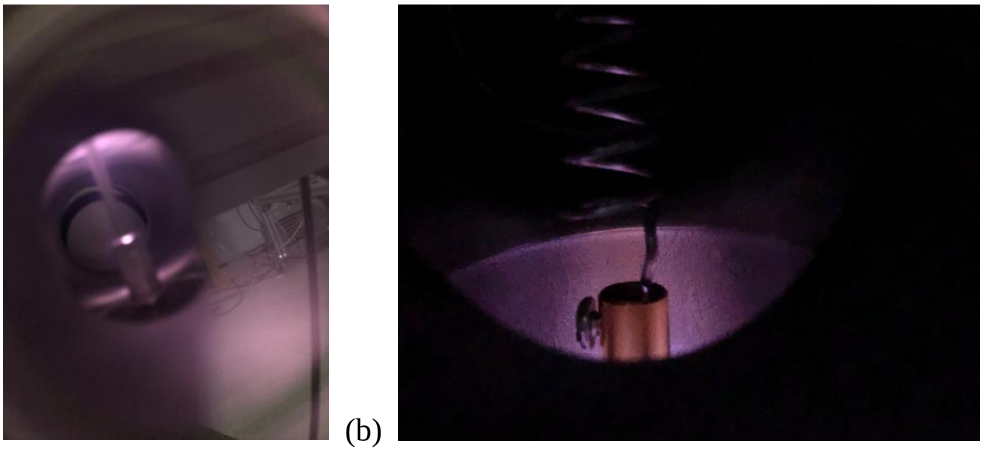

(a) (b)

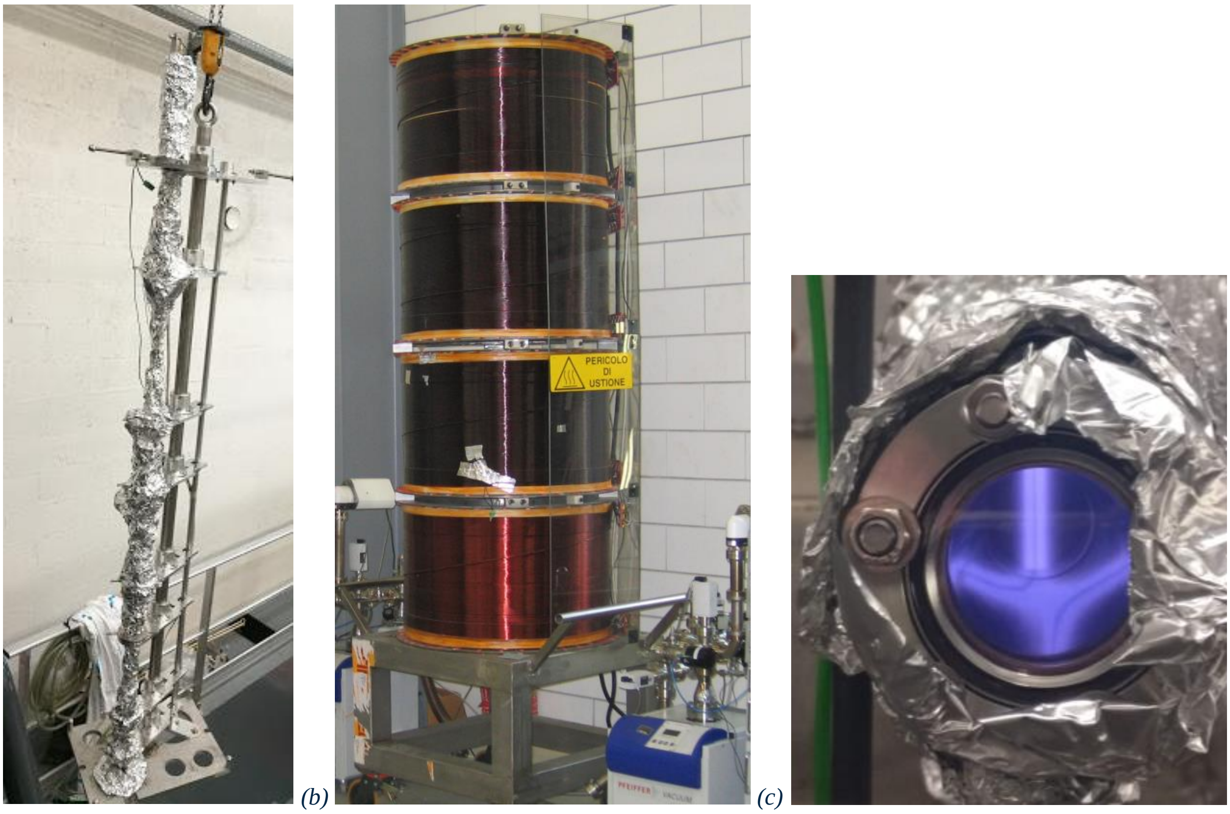
Figure 7: Visual plasma condition view ports (a) the bottom and (b) at the top.

No widness sample was placed in this experiment because the short length of the target as compare to the depsoition tube. The top filament was 950 mm in length and the bottom fialment was 300 mm.

After deposition the sample was tested on NEG coating evaluation facility at UKRI. It has been activated to 160 °C for 24 hours, this temperature is sufficient for partial activation to check whether the NEG film can be activated, but it is insufficient for full activation [18], hence it will not be poisoned and cause any NEG degradation in comparison to a 'virgin' NEG film. After activation, the sticking probability α was measured by pressure ratio method during H_2 and CO injecting, resulting in $\alpha_{H2} = 7\times10^{-3}$ and $\alpha_{H2} = 3\times10^{-2}$, these results are compparable to earlier obtained ones [18].

3.2 Sample for a Soleil test

The NEG-coating deposition facility at SOLEIL comprised of a 2-m-long wide magnetic coil which allows originally to coat SOLEIL's quadrupole vacuum chamber with conventional pumping ports. Any assemblies of 2-m in height can be inserted into the coil for DC magnetron NEG sputtering. The deposition target is traditionally made of three (Ti, Zr and V) twisted wires with 0.5 mm diameter each. After a UHV bakeout, the deposition is classically performed at 100 °C with a Kr discharge gas at a pressure of 1.5×10^{-2} mbar, following CERN legacy recipes. A DC magnetron power supply regulates cathodes currents and applied voltage for constant given power according to processes. Several test samples installed outside of the deposited chamber were deposited together with the vacuum chamber downsizing inner diameters from 63 to 10 mm in stainless steel pipes.



(a) (b) (c)

Figure 8: (a) the 2-m assembly from ID10 mm NEG-coating process at SOLEIL facility, (b) the 2-m coil NEG-deposition bench at SOLEIL facility, (c) a picture of the plasma from the actual ID20 mm NEG-coating process at SOLEIL facility.

Table 2: Deposition parameters for NEG coating at SOLEIL facility.

Magnetron Power	20 W per cathode meter
Current	Variable up to 100 mA
Voltage	Variable up to 550 V
Pulse frequency	DC
Current in magnetic coils	100 A + correction
Deposition time	a few hours with typical 1.5×10^{-5} µm/s
Deposition pressure	$(1–5)\times 10^{-2}$ mbar of Kr support gas
Deposition temperature	100 °C

4 Sample installation on SR beamlines

4.1 Sample installation on SR beamline at DLS

4.1.1 SR beamline

The DLS SR beamline operates as part of the Diamond-II upgrade programme and consists of two sections: a dipole source front-end and an experimental end-station. These are shown in Figs. 9 (a) and (b), respectively.

The front-end is equipped with beam conditioning elements including a defining aperture, horizontal slits, and accompanying X-ray beam diagnostics, and is attached directly to the DLS storage ring.

The end-station is connected to the front-end through a pivoting bellows and this allows for test chamber angles of up to 3.4^{o} (60 mrad). The system consists of two independently pumped volumes capable of supporting a test chamber between them. Movement of the rear pumping volume allows for chambers with lengths between 0.5 and 1 metre with internal diameters of 100 mm. The combined lateral and angular movement range of the end station ensures that the incident X-ray beam illuminates as much of the cooled vessel length as possible. Also present on the end station is a remotely controllable gas injection system with separate pumping arrangement for sticking probability measurements. A suite of cold cathode and RGA gauges are included on both the pumping vessels and the test chamber.

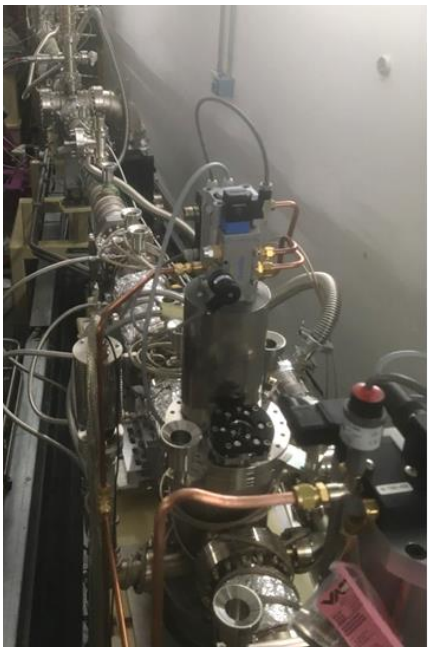
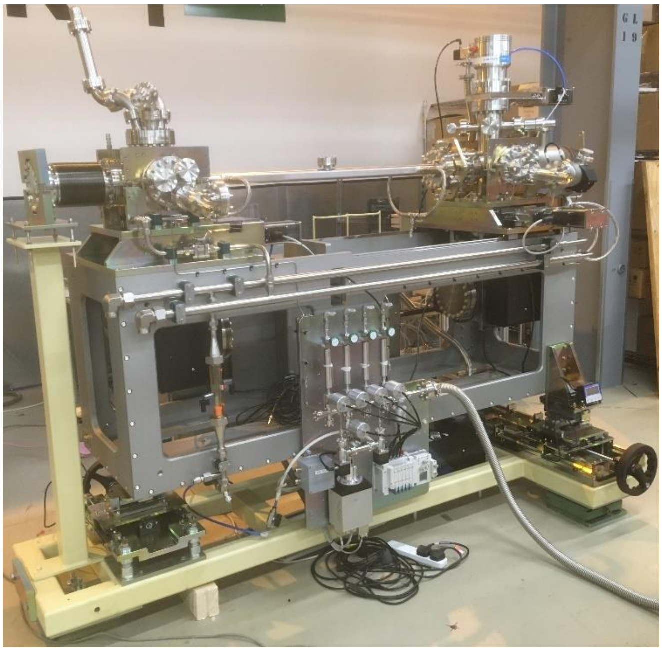
(a)

(b)


Figure 9: (a) an upstream view (towards the dipole source) of the front-end section, (b) the end-station prior to its installation and commissioning..

This arrangement of gauging means that the end-station can be operated in one of two modes to determine the PSD yields, either the transmission method or the three-gauge method [23].

The PSD beamline was commissioned over a period of 6 months in two sections, the front-end was installed during November 2020 and this was followed by the end-station in May 2021. The beamline has been in operation since October 2021 with an uncoated stainless-steel test vessel present on the end-station.

4.1.2 Sample on the SR beamline

As outlined above, a stainless-steel test chamber was NEG coated at UKRI/STFC Daresbury Laboratory and was installed on the DLS SR beamline in March 2022. A picture of the end station with installed chamber is shown in Fig. 10. From the picture, it can be seen that an RGA has been mounted to the central port and, alongside this, a protective conditioning cone assembly has also been included. This cone assembly limits parasitic PSD from the central port and reduces the effects of scattered radiation on the RGA pressure measurements.

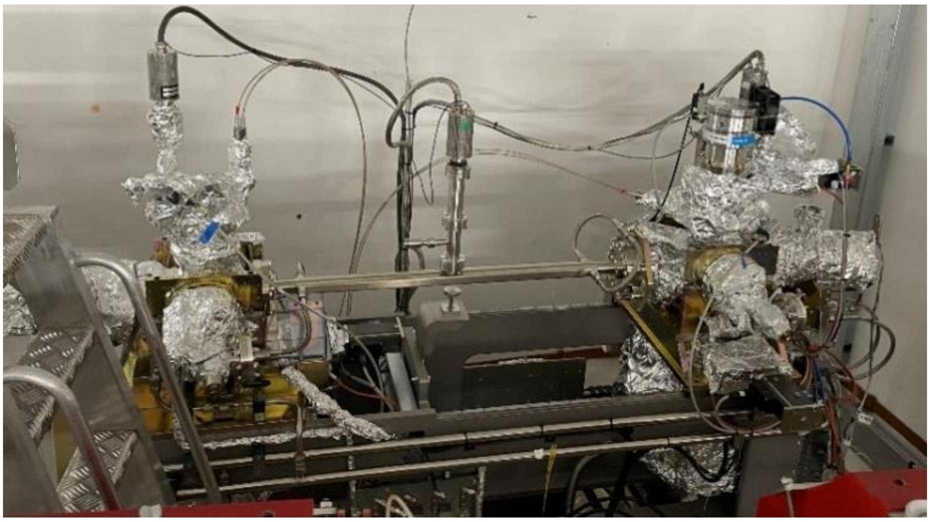


Figure 10: The end-station with the recently installed NEG-coated stainless-steel test chamber. Note the central port is occupied by a conditioning cone assembly and an RGA gauge.

4.2 Sample installation on SR beamline at Soleil

4.2.1 SR beamline

A PSD beamline has been designed and installed on the D08-1 photon exit of the SOLEIL SR, inside of the tunnel wall (see a sketch view of the beamline in Fig. 11. It consists of a pair of moveable diaphragms which precisely adjust in the horizontal plane the photon beam acceptance coming from the bending magnet to the test chambers diameters, and of a large pumping units with two large sputter ion pumps (Starcell®, 400 l/s on N_2) and two sorption getter pumps (Capacitor®, 2000 l/s on H_2) which provide a high pumping speed S (l/s) in front of a conductance C of 28.3 l/s. Behind the conductance C the experimental set up is composed of a calibrated pressure measurement unit P_T (mbar) and the test chambers stand. Since the conductance C is much smaller than the pumping speed of the pumping unit the PSD gas load Q (mbar·l/s) coming from the test chambers can be simplified to C x PT. The evolution of the photon to molecule yield η, indicative of the PSD, is then proportional to the gas load Q divided by the intensity of the incident photons Γ (photons/s).

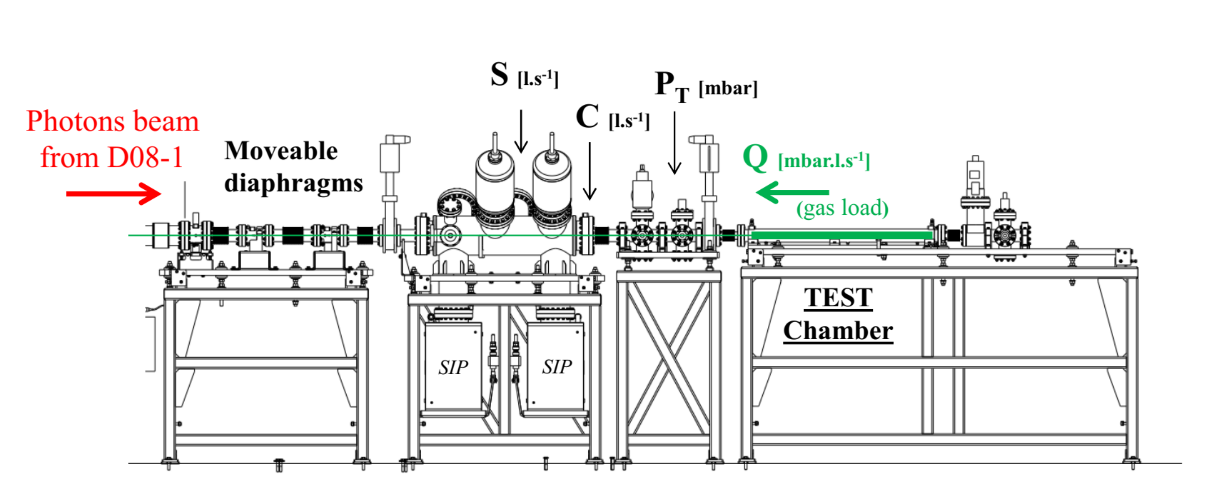


Figure 11: PSD beamline on the D08-1 photon exit of the SOLEIL SR (inside the tunnel wall).

4.2.2 Sample on the SR beamline

In the framework of a collaboration with SAES group, a set of three 1-m long, water cooled vacuum chambers with an inner diameter of 63 mm [DN63], 20 mm [DN20] and 10 mm [DN10] were installed on the PSD beamline. The chambers were previously NEG coated with “state of the art” TiZrV coating of an average thickness of 1 µm.

Before the measurements the experimental set-up was baked-out at 200°C with the NEG coated chambers heated only at 100 °C to desorb water vapour without activating the coating. The incidence angle of the synchrotron light was set to 29.2 mrad for the DN63 diameter (for an opening of the diaphragms of 30 mm) and 7.7 mrad for the DN20 and DN10 diameters (for an opening of the diaphragms of 8 mm) such as the full 1-m length of the wall were exposed to the photon beam.

The PSD yields of the unactivated coatings were first measured during some specific machine shifts to avoid any perturbation of the beamtime delivery to users (2 machine shifts of 8 hours per chamber were needed). The PSD yields reach a maximum at the beginning of the photon beam irradiation (between 3×10^{-2} molecules/photon for DN63 and 9×10^{-3} molecules/photon for DN20 and DN10) then typically decrease by a power function of the dose for all the chambers (see Fig. 12).

After an accumulated photon beam dose around a few ~ 10^{20} photons/m an activation of the NEG coating was performed by heating each of the chambers at 230 °C for 24 h, while keeping the other parts of the beamline at room temperature. As shown on Fig. 11, following these NEG activations the PSD yields sharply decrease by more than a factor of magnitude for all the chambers. The PSD yields reach 1×10^{-4} for DN63, 1×10^{-5} for DN20 and 2×10^{-5} molecules/photon for DN10 and are obtained only after a few minutes of photon beam bombardment. As a comparison, these values of total desorption are usually obtained with unactivated NEG coating or standard materials after weeks of synchrotron light commissioning. Also, the discrepancy between the evolutions of the PSD yields before and after activation for DN63, DN20 and DN10 can be explained by the NEG coating intrinsic properties after NEG deposition i.e. the initial sorption capacity and initial sticking probability. These properties can significantly differ from one chamber to another but can be precisely characterized in the vacuum laboratory at SOLEIL on transmission method test benches.

Finally, the behaviour of the PSD yields after NEG activation for vacuum chambers diameters between DN63 and DN10 clearly indicates that there are no downscaling issues from a pumping point of view for a synchrotron light commissioning. It also demonstrates that a SR vacuum system upgrade based on a full NEG coated ring can be pursued even with a characteristic size of the vacuum chambers as low as 12 mm in diameter.

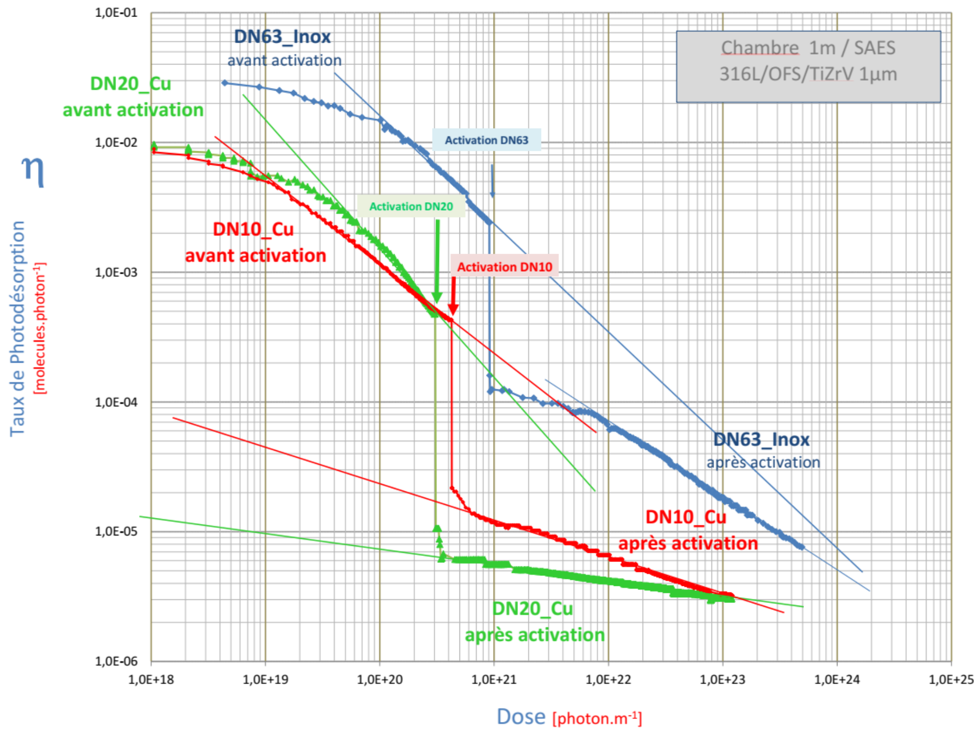


Figure 12: Evolution of the PSD yield before and after NEG coating activation as a function of the photon beam dose for three 1 meter long water cooled chambers from SAES group. The chambers have an inside diameter of 63 mm (DN63), 20 mm (DN20) and 10 mm (DN10). They are NEG coated with 1µm of TiZrV. The activation of the NEG coating is performed for all the chambers at 230 °C for 24h.

5 Future plans / Conclusion / relation to other IFAST work

5.1 Conclusion

Report is related to achieving I.FAST Milestone 48 (First NEG coated samples are installed on SR beamline at DLS and Soleil). A new dedicated SR beamlines was built and tested at DLS, a sample coated with NEG at UKRI/STFC has been installed and ready for a test immediately after restarting DLS operation (after scheduled shutdown) from mid-April. An existing dedicated SR beamlines at Soleil Synchrotron was recommissioned and tested with 3 samples coated with NEG at SAES Getters. Thus, the I.FAST Milestone 48 (First NEG coated samples are installed on SR beamline at DLS and Soleil) has been met.

5.2 Future plans / relation to other IFAST work

After testing the beamlines with installed NEG coated samples, the obtained data must be analysed and discussed. After delivering new copper samples, two of them will be coated at UKRI with a single metal (Zr) film and installed at DLS and Soleil beamlines during shutdowns in August 2022. The PSD yields from the samples will be studied in various condition until full degradation of the NEG film. The following samples will be coated and installed for the testing after that.

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Annex: Glossary

Acronym	Definition
AFM	Atomic Force Microscope
CERN	European Council for Nuclear Research, Geneva, Switzerland
DESY	
DLS	Diamond Light Source, Didcot, Oxfordshire, UK
EDS	Energy Dispersive X-ray Spectrometry
EP	Electropolishing
HIPIMS	High power impulse magnetron sputtering
OFE	Oxygen Free Electronic copper
OFHC	Oxygen-Free High thermal Conductivity copper
NEG	Non-evaporable getter
SEM	Scanning Electron Microscope
Soleil	French national synchrotron, stands for French words : Source Optimisée de Lumière d'Energie Intermédiaire du LURE
SR	Synchrotron Radiation
UKRI/STFC/DL	United Kingdom Research and Innovation / Science and Technology Facilities Council / Daresbury Laboratory
TF	Thin films
XRD	X-Ray Diffraction