MEDSI 2014 Conference

Melbourne, Australia - October 2014

Design of a new detector tube for wide, small and ultra small-angle X-ray scattering

Joachim Léonardon, Pierre Van Vaerenbergh, Theyencheri Narayanan, Michael Sztucki, Peter Boesecke, Jacques Gorini, Serge Guillet[#], Paul Berkvens, Yves Dabin, Loys Goirand, John Morse, David Martin

ESRF-The European Synchrotron

CS40220, 38043 Grenoble Cedex 9, France

joachim.leonardon@esrf.fr; vanvaer@esrf.fr; narayan@esrf.fr; sztucki@esrf.fr; boesecke@esrf.fr; gorini@esrf.fr; berkvens@esrf.fr; dabin@esrf.fr; goirand@esrf.fr; morse@esrf.fr; martin@esrf.fr

Pedro Noguera, Jon Osoro

AVS-Added Value Industrial Engineering Solutions S.L.

Pol. Ind. Sigma Xixilion Kalea 2, Bajo Pabellon 10, 20870 Elgoibar, Gipuzkoa, Spain

pnoguera@a-v-s.es; josoro@a-v-s.es

Abstract - In July 2014, the ESRF reopened the upgraded ID02 beamline which offers a unique, multipurpose small-angle, wide-angle and ultra small-angle X-ray scattering (SAXS, WAXS, USAXS, respectively) facility. The key technical component of this beamline is a stainless steel detector flight tube 34 m long and 2 m diameter comprising 5 sections each of 6.7 m length. Three different SAXS detectors are housed inside a sealed, motorized wagon which travels along a rail system inside this tube. This arrangement allows automated change of the sample-to-detector distance over the range of 0.5 m to 30 m in SAXS/USAXS experiments. All the tube joints, vacuum feed-throughs, doors and cable chicanes satisfy radiation protection regulations, so that the interlocked flight tube provides all necessary radiation shielding without requiring a surrounding lead hutch. The interior of the wagon remains at atmospheric pressure while the 100 m^3 volume tube is evacuated to 10^{-2} mbar in less than 2 hrs using an industrial dry screw pump. A robust personnel safety system controls the access to the flight tube preventing any accidental trapping of personnel inside the tube, and inhibits vacuum pump operation and wagon motion until safe conditions are established. The precision engineering and accuracy of the alignment of the flight tube segments and wagon guide rails restrict the parasitic movements (circle of confusion) of the wagon within ±0.3 mm of the ideal axis along the entire 30 m travel. A novel SAXS beamstop system installed on the front flange of the wagon enables monitoring of the X-ray beam intensity in addition to blocking the primary beam, and an automated insertion of selected masks behind the primary beamstop. This contribution will present the key mechanical design features, assembly protocols and operational procedures of this new detector tube.

Keywords : Detector – Tube – SAXS – WAXS – USAXS – Beamstop

[#] Present address : SLAC National Accelerator Laboratory, 2575 Sand Hill Road, Menlo Park, CA 94025, United-States, sguillet@SLAC.Stanford.EDU.

Introduction

In 2009, the European Synchrotron Radiation Facility (ESRF) launched an ambitious upgrade program (ESRF, 2007). The phase I of this program included an extension of one part of the Experimental Hall that allowed installation of long beamlines and full upgrade of 8 beamlines over a period of 7 years (2009-2015). One of these beamline projects was UPBL9a that involved a major upgrade of ID02 beamline which is dedicated to small-angle X-ray scattering (SAXS) and related techniques. SAXS is a well established method to probe the structure and dynamics of non-crystalline materials over the nanometric size scales (T. Narayanan, 2014). The primary goal of the UPBL9a project was to push the limit of SAXS to ultra low angles of microradian ranges. This is realized via a combination of new focusing optics which preserves the source brilliance, high degree of collimation in the horizontal direction and high angular resolution detection of the SAXS intensities.

This paper concerns the design of the SAXS detector tube to cover a useful scattering angular range from about 10 microradian to 0.2 radian. This wide angular range is covered by varying the sample-to-detector distance from 0.5 m to 30 m. The detector tube facilitates an easy change of sample-to-detector distance very precisely and reproducibly without breaking the vacuum inside the tube. In addition, several detectors are required to obtain optimum angular resolution, wide angular range at a given detector position and time resolution. The detector tube houses these detectors and enables their rapid exchange as desired. All these requirements resulted in a large detector tube of 34 m long and 2 m diameter. The addition of a wide angle detector extends the scattering/diffraction angular range to about a radian. The upgraded ID02 beamline is open to the user community since July 2014, and provides a unique multipurpose small-angle, wide-angle and ultra small-angle X-ray scattering (SAXS/WAXS/USAXS) facility. The combination of these three techniques covers nominal real space dimensions from 1 Å to 10 microns using 12.4 keV X-rays.

Structure of the flight tube

Figure 1 depicts the 34 m SAXS tube which is made of 5 sections of 6.74 m long, a flat front flange and a torispherical rear flange all built in stainless steel (304L). The number of sections had to be minimum in order to limit the number of flanges which are critical points of the tube due to gaskets, extra weight, and stress induced into the material. At the same time, the weight of each section should not exceed the lifting capacity of the overhead crane, which was 6 200 kg, available for the unloading from the delivery trucks. Each section was made with a wall thickness of 8 mm, 6 circular ribs of 20 mm thick and 52 mm high; at each end 2 flanges 60 mm thick and 82 mm high with a gasket groove and 6 feet supports. This resulted in a net weight of the sections between 5 500 kg with lateral door and 5 100 kg without door.

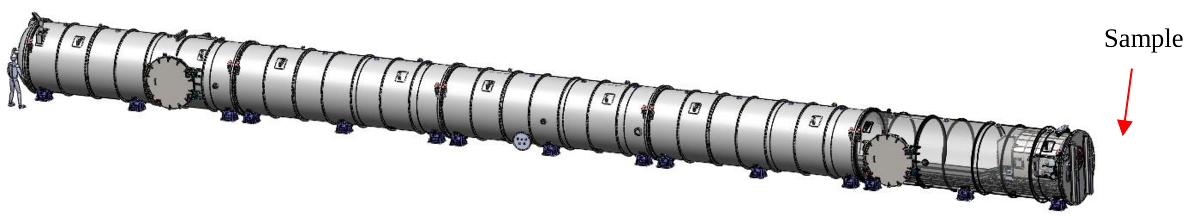

Figure 1: Overview of the 3D model of the SAXS Tube (transparent first section).

The end flange has torispherical shape as a natural design for a pressure vessel. For the front flange, the constraint of minimum sample-to-detector distance of 500 mm was fulfilled by using a 45 mm thick flat flange. Two large I beams (200 mm high, 150 mm wide, 1800 mm long) reinforce the flat flange on each side of the X-ray beam aperture and reduce the flange deflection once the tube is evacuated.

Frame and tube supports

A high stiff structure has been developed in order to fix the rails and to reach the straightness within a disk of confusion of diameter 3 mm (initial conservative goal). This structure is made by 5 sections, identical length to the tube sections, fixed one to the other giving a 30 m long high stiff structure as shown in Figure 2a.

Once the tube is evacuated, this configuration limits the parasitic movement of the frame due to the deformation of the wall during the depressurization. Inside each section of the tube, 5 pairs of welded brackets are used for fixing the rigid frame which also supports the rails as indicated in Figure 2b.

The 6 feet of each section are bolted to the slab. A 4 cm grouting (SICA-Grout 295) has been poured between the slab and the feet as shown in Figure 3. The foot has 4 screws M30 for aligning and keeping the section but only the 4 first feet of the first section are immobilized in the X direction. Thus, most of the tube can expand in X direction due to temperature variation without introducing constraints inside the material or deteriorating the rail straightness.

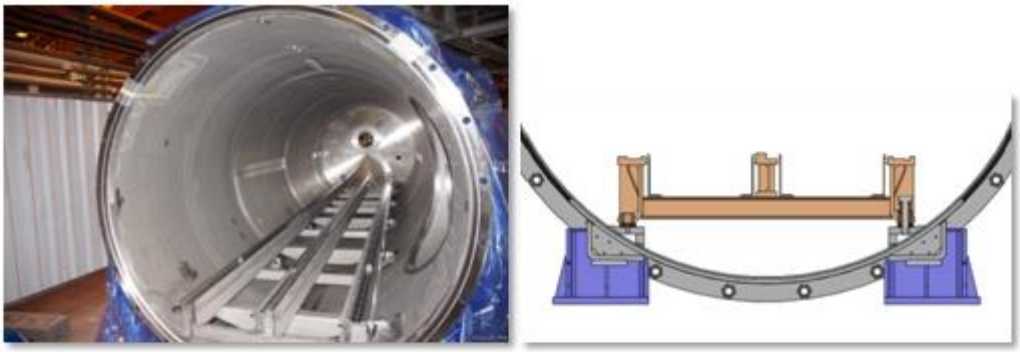

Figure 2 a, b: (a) Stiff structure section supporting the rails. (b) Fixation of the structure at the vertical position of the feet.

Figure 3: Feet of the tube.

Concrete slab

The SAXS tube was installed in the two existing Experimental Halls and partly located below the intermediate office building as indicated in Figure 4. The front part of the tube is located in the Experimental Hall 1 (built in 1992), the rear part of the tube lies on the new Experimental Hall Belledonne slab. However the middle of the tube sits on the old free way and slab of the office building. In order to avoid the instabilities that may have been induced by these weak slabs, a new slab of 24.5 m long, 2.2 m wide and 0.35 m thick was built to support the tube along the sections in the old building.

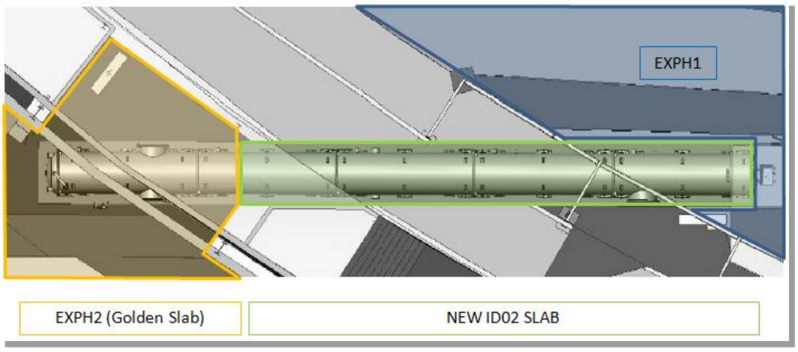

Figure 4: A dedicated concrete slab to support the tube in the old building and dead zone between the Experimental Halls.

Structural engineering analyses

All the tube associated elements (wall thickness, ribs, feet, flanges) have been dimensioned in compliance with CODAP 2010, which is French code for the construction of pressure vessels to comply with the requirements of the European Directive 97/23/CE (SNCT, 2010) and the EN ISO 13445 (AFNOR, 2006) by taking into account the proper weight of the elements, an external pressure of 1 bar and a temperature variation of 10 °C between the two extremities of the tube. According to CODAP 2010, for 304L stainless steel, the maximum stress must be lower than 136 MPa. As a result of the calculations, the first damages could appear locally at 1.55 bar but without risk of buckling.

In order to limit the parasitic movements of the detectors, the structural deformations and the ground settlement deformations were calculated.

A special attention was paid to the optimization of the carriage with the aims of limiting the parasitic movements and the risks of balls breakages due to hyperstatism. Thus the risk for failure of the carriage is as low as possible which should ensure a mean-time between failures as long as possible.

The flanges were dimensioned for the use of metal gaskets between the sections.

Dynamic analyses allowed us to determine the natural frequency of the tube (8 Hz), its behavior in case of an earthquake (avoid collapsing), and the effect of rupturing of a window (maximum speed of suction: 720 $m.s^{-1}$).

The settlement was a big issue due to the non-acceptable behavior of the already existing slabs and the type of ground at the ESRF site (shingle). Therefore a new dedicated slab was analyzed and dimensioned to limit ground settlement and to avoid tube buckling.

The final calculations were made by the subcontractor AVS, using the MSC programs (PATRAN 2011: meshing and interpretation of results, MARC 2012: solver for non linear calculations and NASTRAN 2012: solver for linear calculations).

Vacuum tightness

The sections were assembled by placing in contact the end flanges and tightening 24 M30 bolts with a torque of 1 200 Nm. The vacuum tightness is guaranteed by means of metal gaskets. The choice of metal gaskets came from the need of having a maintenance free system while the polymer gaskets have only recommended working life of 8 years. The tunnel shape environment around the tube, without overhead crane access, makes very difficult the changing of gasket between two sections if a leak would occur on such polymer gasket. Nevertheless, polymer gaskets were used for front and rear flanges since they may need to be opened occasionally and can be handled by overhead crane.

Installation

The whole tube was first assembled at AVS workshop. For shipment, the tube was disassembled and each section was carried and delivered with the associated pre-aligned frame section and rails fixed inside. For installation no crane was used as the 4 first sections are located in a corridor, like a tunnel 3 m wide and 2.5 m high, between the old and new Experimental Halls. These sections were carried on by rolling pads, pulled with a cable and pushed by hands as illustrated Figure 5.

Figure 5: Installation of tube sections in the corridor.

Alignment and survey

The installation at the ESRF started with the alignment of the first section with respect to the X-ray beam thanks to laser trackers. Subsequent sections were installed and aligned with respect to the previous ones and along the X-ray beam axis. A fine alignment of the master rail was performed and then the slave rail was adjusted with respect to the master rail.

The final survey of the parasitic movement of the wagon in air was carried out using two laser trackers (AT401), which gave a maximum deviation of 0.8 mm (repeat $1.8\text{x}10^{-5}$ m) in horizontal and 0.6 mm (repeat $2.7\text{x}10^{-5}$ m) in vertical. In vacuum, the diameter of the disk confusion was measured to be lesser within 0.6 mm over 30 m stroke, as revealed by a high resolution X-ray beam viewer.

Since March 2014 a long term movement survey (by means of hydrostatic leveling systems and laser trackers campaigns) is in progress with the aim of better evaluating the tube behavior as summarized in Table 1. For the moment, repeated cycles of operation and the timescale have no significant impact on the mechanical performance of the tube.

Table 1: SAXS tube long term movements observed so far.

Operation	Direction	Peak to peak movement (µm)	Timescale
Wagon	Vertical (z)	65	Seconds or minutes
Vacuum	Vertical (z)	130	2 hours
Vacuum shortening	Longitudinal (x)	600	2 hours
Thermal/Diurnal	Vertical (z)	20	1 day
Slab movements	Vertical (z)	160	Weeks to months

The wagon

The wagon is an assembly made of a cabin, a translation table (horizontal Y and vertical Z) and a carriage as shown in Figure 6a. Under normal operation, interior of the cabin is at atmospheric pressure, and outside of is vacuum. Therefore the cabin structure is under pressure (1 bar) once the tube is evacuated. In order to limit the deflection of structure, the cabin had to be made of stainless steel with a base plate (40 mm thick), two side walls (20 mm thick with 3 ribs), a rear wall (30 mm thick with 7 ribs) and an arch shaped roof like the tube shape reinforced with a square mesh of ribs. The front of the cabin is closed by a bolted flange (30 mm thick) airtight thanks to a polymer gasket. The accesses to the inner of the cabin can be made through the main access door (on hinges, 900x550 mm) at the rear side or by five secondary doors, one at the front and two on each lateral side. On the front flange, the X-ray transparent window is made of fibrous carbon sheet (400 µm thick, 388 mm in diameter) which is glued with Araldite and pressed with a bolted ring on a stainless steel flange as depicted in Figure 6b. The cabin can be adjusted with respect to the X-ray beam in the horizontal Y and vertical Z directions by the means of a translation table (YZ1) installed between the cabin on top and the carriage on bottom. In order to avoid any wedging of the translation table, the four connections between the cabin and the table are spherical. The YZ1 translation table allows, if necessary, repositioning of the center of the detector within a disk of confusion of 100 µm diameter centered on the X-ray beam over the 30m stroke.

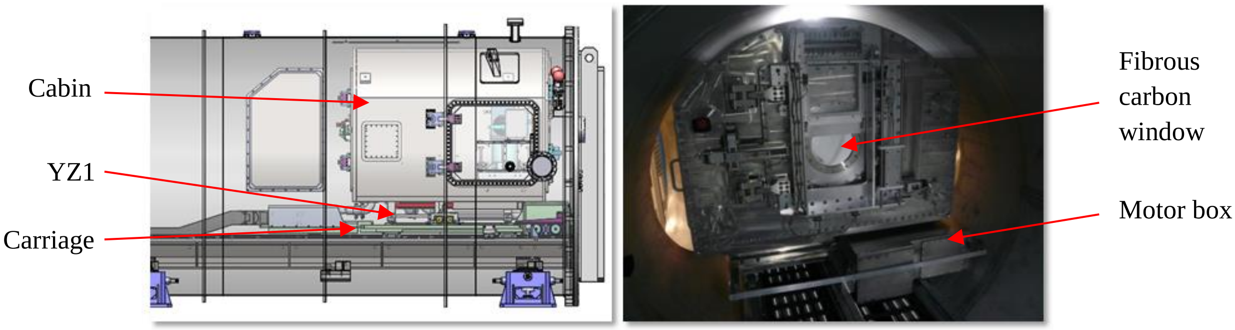

Figure 6a and b: (a) Wagon made of carriage, YZ1 translation table and cabin.
(b) Fibrous carbon window and beamstop fixed on the front flange of the wagon and the motor box.

Below the YZ1 translation table, the base frame of the wagon is the carriage. Four pads fixed under the carriage allow the wagon to translate along a stroke of 30 m. The wagon is actuated by a brushless motor (Wittenstein TPM050S-091M-6PB1-130A-W1) housed in a vacuum tight box fixed in the front of the carriage. The motor is connected to a double pinions system shown in Figure 7a, which gears on a central rack. By acting on two opposite faces of rack teeth, the two pinions allow the backlash to be reduced. For the accurate position control of the wagon along the 30 m stroke, an absolute encoder (Baumer GM400.A11A102) is connected to the pinion. The motor box is connected to the cabin by two flexible tubes (KF50, 750 mm length). The motor is cooled by constant flow of compressed air pushing the hot air of the motor enclosure through the flexible tubes. Many cables (46 cables and 6 dual optical fibers) and 9 hoses are connected to various equipment inside the wagon to outside supplies. These cables and hoses are all enclosed inside 10 flexible corrugated stainless steel tubes. The flexible tubes are split in two groups of four tubes 50 mm diameter and one tube of 30mm diameter as indicated in Figure 7b. Each group is carried by a cable chain connected between a fix point close to the horizontal cable exit tube 406 mm diameter (in the middle of the tube) and the rear of the carrier. The cables are drawn into the cabin through 10 short flexible tubes. Thus the strength of the flexible tubes due to their expansion under vacuum (about 1 %) is applied to the frame and not to the cabin. The moment of force due to the extension of short flexible tubes is negligible as compared to the weight of the cabin (3 000 kg).

Figure 7 a and b: (a) Double pinion of the motor box. (b) Flexible corrugated tubes inside two cable chains.

Equipment inside the cabin

Inside the cabin, three SAXS detectors (Rayonix MX170, Frelon 4M and Pilatus 300K) and a beam viewer are mounted on a translation table YZ2 as depicted in Figure 8a. This table allows the user to select the desired detector and center it with respect to the fibrous carbon window (horizontal stroke: ±320 mm). Moreover YZ2 permits to off center the detector by moving vertically by ±60 mm. The power supplies and vacuum system for the detectors and other control accessories are housed inside the cabin. The temperature inside the cabin is maintained at 26 °C by a fanned air/water heat exchanger. The arrangement of the rack, YZ2 table and the cable trays inside the cabin allows a person to enter inside the cabin for maintenance and installation as indicated in Figure 8b.

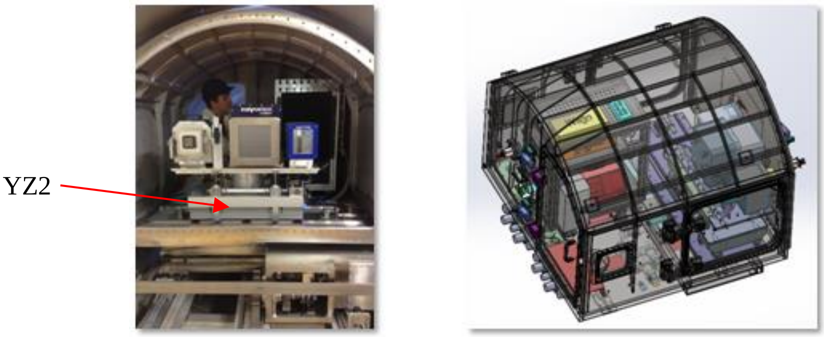

Figure 8 a and b: (a) Frelon 4M, beam viewer, Rayonix MX170, Pilatus 300K detectors on YZ2 translation table. (b) Tight packing of equipment inside the cabin.

Beamstop

In order to block the direct beam and facilitate the monitoring of the direct beam intensity, a novel beamstop system is installed in front of the X-ray window of the wagon. It is made of two independent systems as illustrated in Figure 9a. The primary beamstop (BS1) and the secondary beamstop (BS2), are bolted on the front square door as indicated in Figure 6b. The primary beamstop is a lead block of 2 mm high, 5 mm wide and 4 mm thick in which a silicon *p-i-n* diode is embedded. The lead block is anchored on a 145 mm long titanium tube of diameter 1.6 mm which also carries the leads from the photodiode. The tube is fixed on an integrated YZ translation stage (vertical stroke ±21 mm and horizontal stroke ±111 mm) allowing to even displace BS1 out of the X-ray window. The secondary beamstop (BS2) is a group of 6 independent beamstops of various shapes and sizes from 1 mm to 12 mm. Each beamstop is stuck on a thin PET film which is glued on a thin aluminium frame. All 6 aluminium frames are identical in size 300 mm high, 290 mm wide and 3 mm thick. An elevator carries them up to the parking position and brings down to the measurement position when desired. The frames are kept at the parking position by means of 12 magnetic actuators (2 by frame in symmetrical position with respect to the middle). Each aluminum frame has two hooks which are bolted on the top side. The hooks are designed in such a way as to maintain the frames automatically without electrical activation of the actuators. The actuators keep the frames in parking position by inserting the actuator arms in a horizontal groove machined in the hooks as shown in Figure 9b.

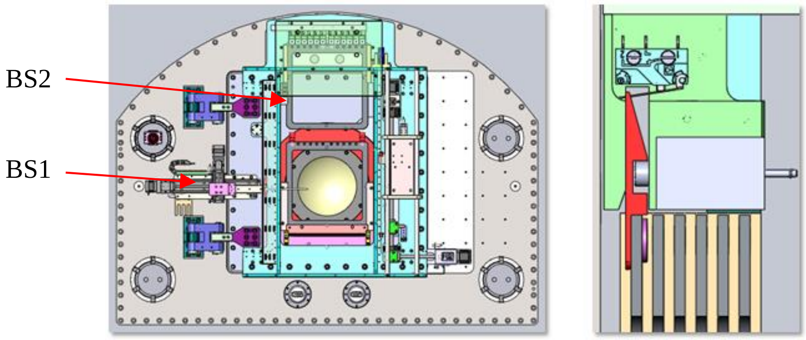

Figure 9 a and b: (a) The primary beamstop (BS1) and the group of 6 frames of the secondary beamstop around the fibrous carbon window (BS2). (b) Side cut of the frame fixing hook system.

Above these horizontal grooves, the hooks have an inclined plane which pushes the actuator arm when the frame is carried up to the parking position. To change the frame, the elevator moves up to the highest position. Then the selected frame is released by switching on the corresponding magnetic actuators and it is then carried down to the measurement position. In order to prevent the frames falling down due to an accidental activation of the actuators, a contact switch opens the electrical circuit of the magnetic actuator power supply when the elevator reaches 20 mm below the parking position. The elevator is made of a cassette with 6 vertical slots in which the selected frames are inserted and carried. The number of simultaneously selected frames is from zero to six. The global translations Y (±20 mm stroke) and Z (375 mm stroke) are guided with thin pads and rails system and actuated by one stepper motor for each. In order to limit the size and the warming up of the motor, a counterweight, suspended by a cable, balances the weight of the elevator.

Detector support and WAXS cone

The Wide Angle X-Scattering (WAXS) detector Rayonix LX170 is located outside the detector tube. The detector support is fixed on the tube front flange. It translates on rail and carries the WAXS detector from the working position (lowest position, z = 0 mm), to the parking position (highest position, z = 487 mm) as shown in Figure 10 a and b, respectively. The elevation is done manually and balanced by a counterweight.

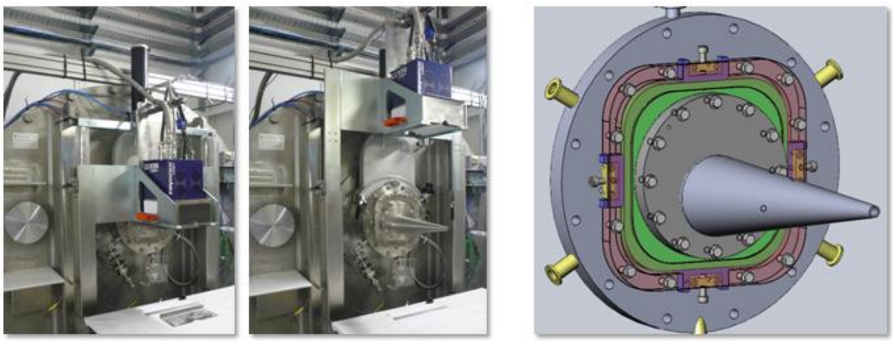

Figure 10 a, b and c: (a) WAXS detector at working position. (b) WAXS detector at parking position. (c) WAXS cone fixed on the adjustable flange.

The bottom side of the Rayonix LX170 has a groove cut which fits closely the WAXS cone. The cone is 451 mm long with a cone head aperture of 12 mm diameter and a base aperture of 103 mm diameter welded of a 220 mm diameter flange. The cone axis has an angle of 3.15° with respect to the beam axis. This angle allows the WAXS detector to be lowered at the maximum and gives the widest angle in the bottom side for scattered X rays, taking into account that the cone is bolted on an adjustable flange. All the different cones are easily replaced thanks to an identical base flange. The vacuum isolation is made by a 12 mm diameter mica window, which is glued on the cone head aperture and the cone base flange is fastened on to the adjustable flange with a polymer gasket in between. The flange is manually adjustable in Y (±5 mm) and Z (±5 mm) in order to align the cone entrance to the X-ray beam axis by means of 4 screws as indicated in Figure 10c.

Radiation protection

ESRF is a facility where staff and visitors are not exposed to ionizing radiation. That means that the cumulative exposed dose has to be lower than 1 mSv/year. In order to comply with this limit over the operating X-ray energy range of the beamline, the tube wall thickness has to be at least 6 mm. We used 8 mm for structural reasons. All flanges, doors and apertures were chicaned by means of specific machining (flanges between sections, rear flange, lateral doors) or additional local shielding (e.g. exits of the flexible tubes carrying cables and hoses, connection to the vacuum pump, vacuum gauges, feed-throughs for emergency stops and lights, etc.). Therefore the tube provides all necessary radiation shielding without a surrounding lead hutch.

PSS

The tube is equipped with a robust Personnel Safety System (PSS) which ensures that an accidental trapping of a person inside the tube does not occur under any circumstances. The custom system is developed from the standard PSS installed for lead hutches at the ESRF. The PSS inhibits the operation of the vacuum pump and wagon movement until the safe conditions are established by a specific search procedure. The access to the tube is restricted to selected number of staff and certain mandatory procedures need to be followed for any interventions inside the tube. The PSS search procedure involves positioning of the wagon at an intermediate position and two authorized persons to perform the search simultaneously from either ends of the tube. The tube interior is well illuminated by 10 LED panels. 8 emergency stops are installed on the inner walls of the tube and both sides of the wagon. In addition, the wagon is equipped with an anti-collision system. Once the PSS is correctly interlocked, the vacuum pump and the wagon motor control are enabled.

CE Certification

The French labour law specifies to CE certify all machines used by workers. Therefore the compilation of the incorporation certificate from the sub-contractor, a risk assessment in compliance with the standards ISO 14121-1 (AFNOR, 2007) and ISO 14121-2 (AFNOR, 2008), a user safety manual and the structural engineering analyses, allows the SAXS tube to be CE certified on 12th June 2014.

Conclusion

The SAXS detector tube at UPBL9a was a collaborative project involving participation of more than 150 people. Thanks to the sub-contractors AVS and Cadinox, the obtained mechanical performance of less than 0.6 mm parasitic movement is five times better than the design goal. The facility is quick to operate since it reaches working vacuum after 60 min of pumping. It is a safe equipment in compliance with the legislation, which is validated by the CE certification. The upgraded ID02 beamline is in operation since July 2014 and it meets the original project goals. Now, a long term movement survey is in progress in order to better understand the structural behavior. A closed-loop control for the wagon alignment will be installed in order to improve the disk of confusion to within 200 μm.

Acknowledgments

We gratefully acknowledge the contribution of M. Bereciartua, J. Fortuna, M. Izaguirre from AVS, J. Brechet, V. Landuré, F. Monnier, S. Ricot, D. Avogadro, G. Gatta, B. Perret, O. Hignette, S. Arnaud, P. Gamond, P.S. Mackrill, T. Marchial, F. Pegot, S. Rigotti, J.M. Taormina, F. Torrecillas, J.M. Clement, H. Gonzalez, M. Kocsis, J.C. Labiche, J.J. Thévenin, L. Claustre, F. Sever, P. Guerin, M. Garrec, M. Hahn, L. Michel-Villaz, H. Pedroso Marques from ESRF and C. Amrouni, L. Didier, A. Perillo-Marconne from ILL to this project.

References

AFNOR, EN ISO 13445, AFNOR 2006

AFNOR, EN ISO 14121-1, AFNOR 2007 2eme tirage 2009-03-P

AFNOR, ISO/TR 14121-2, AFNOR 2008 1st issue 2008-02-P

ESRF, The “Purple Book”, Science and Technology Programme 2008-2017, ESRF (2007)

T. Narayanan, Small-angle scattering, in Structure from Diffraction Methods, Eds. D.W. Bruce, D. O'Hare and R.I. Walton, p 259-324 (J. Wiley & Sons, 2014)

SNCT Publications, CODAP Division 1 Code de construction des Appareils à Pression non soumis à l’action de la flamme, Édition 2010 (Rév. 06/14)