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A differential pumping system to deliver windowless VUV photons at atmospheric pressure

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In order to deliver VUV (vacuum ultraviolet) photons under atmospheric pressure conditions, a differential pumping system has been built on the DISCO beamline at the SOLEIL synchrotron radiation facility. The system is made of four stages and is 840 mm long. The conductance-limiting body has been designed to allow practicable optical alignment. VUV transmission of the system was tested under air, nitrogen, argon and neon, and photons could be delivered down to 60 nm (20 eV).

Keywords: differential pumping; vacuum ultraviolet (VUV) photons.

1. Introduction

Vacuum ultraviolet (VUV) is usually defined as the portion of the electromagnetic spectrum for which UV light has to be transported and manipulated under vacuum (Schumann, 1903). Indeed, VUV starts at 180 nm where atmospheric air and especially molecular oxygen strongly absorb and thus cut off this part of the spectrum (Ladenburg & Van Voorhis, 1933; Watanabe *et al.*, 1953). In order to reach shorter wavelengths, experimental set-ups are usually flushed with pure nitrogen, as often performed for circular dichroism experiments (Johnson, 1988). Nevertheless, below 110 nm LiF and MgF_2 windows are not transparent and VUV may only be delivered to the sample under high vacuum. This somewhat limits the use of photons below 110 nm to materials that can withstand a high vacuum. Unfortunately, many samples such as liquids or living biological materials cannot be placed under reduced pressure. Nevertheless, rare gases are transparent to VUV. Below its ionization threshold at 15.76 eV (78.67 nm), argon is essentially transparent, aside a few discrete transitions at 11.624, 11.828 and 14.3 eV (Chan *et al.*, 1992). Similarly, neon may be used up to its ionization threshold at 21.56 eV (57.5 nm), although it shows very strong discrete absorptions at 16.85 eV (73.6 nm) and around 19.75 and 20.1 eV (Chan *et al.*, 1992). Helium is ionized at 24.59 eV (50.4 nm) and starts absorbing at 21.218 eV (Chan *et al.*, 1991).

The need for the removal of vacuum windows has been recognized in the hard X-ray range (Gog *et al.*, 2007). These workers have reported the concept of a windowless transition from the atmosphere to a high vacuum for millimeter-sized beams for hard X-rays experiments. Unfortunately, their system was not tested on a beamline and the alignment issue was not addressed. Beside this first report of a millimeter-sized aperture to the atmosphere, a few other attempts have been reported for micro-X-ray beams through differentially pumped capillaries (Nebiki *et al.*, 2006) or using a plasma arc (Hershcovitch, 1998; Pinkoski *et al.*, 2001). Recently, Tamenori reported a compact four-elements differential pumping system with a millimeter-sized aperture for windowless experiments in the soft X-rays regime under a helium atmosphere (Tamenori, 2010). For the first time, such a system was tested on a beamline and its optical transmission was measured.

It is worth noting that for both soft and hard X-ray photon energy ranges a few materials may be used as windows for a physical separation between the sample chamber and the beamline vacuum. In the VUV regime below the LiF cut-off, all materials are opaque and nothing may be used as a window.

Hence, the only way to deliver those photons at atmospheric pressure is to use a windowless connection between the sample placed under a rare-gas atmosphere and the beamline through differential pumping.

We describe here a system designed for windowless VUV experiments, which is mounted on the atmospheric pressure experiments branch (APEX) of the DISCO beamline at the SOLEIL synchrotron radiation facility (Giuliani *et al.*, 2009). This system makes the transition from neon or argon atmospheres to the beamline vacuum. It was challenging to create such a system for a VUV beamline based on a bending magnet. Indeed, even on third-generation machines the divergence of the beam requires quite large apertures to accommodate the beam, thus resulting in large conductance.

2. Consideration of the differential pumping assembly

A differential pumping system is used to accommodate the pressure difference between the two parts which have to be connected together. The principle behind differential pumping assemblies comes down to reduction of the conductance between several stages using holes or small pipes. As a consequence, the performance of differential pumping strongly depends on the aperture size and length of conductance-limiting pipes. In the present case the system has to accommodate atmospheric pressure on one side and an ultrahigh vacuum for the beamline to operate, which is 10^{-5} Pa. Hence, the system must reduce the pressure by at least nine orders of magnitude.

As noticed by Tamenori (2010), narrower and longer pipes are not convenient for this purpose, even though modern synchrotron radiation sources produce small diverging beams. Divergence is especially critical when dealing with a bending-magnet-based VUV beamline.

A tremendous amount of literature exists on gas flow in vacuum systems (O'Hanlon, 2003; Rozanov & Hablanian, 2002). Moreover, previous work describing atmospheric-pressure differential pumping systems has provided summaries of the theory applied to this case (Tamenori, 2010). We shall therefore not discuss the theory in the following but restrict ourselves to the qualitative description of prerequisites and functioning.

The principle of the system is summarized in Fig. 1. It is based on the assembly of evacuated chambers separated from each other by conductance-limiting parts. Synchrotron radiation has to be propagated through these parts.

The system consists of four stages in series. Stage 1 contains the orifice, which was at atmospheric pressure and was evacuated by two Varian Triscroll 600 pumps of 25 m^3 h^{-1} pumping speed. Stage 2 was pumped by one Varian Triscroll 600 pump working at a pumping speed of 25 m^3 h^{-1} pumping speed. In stage 3 a water-cooled Pfeiffer turbo pump TMU521 backed by a Varian Triscroll 300 was used. The pumping speed was 500 L s^{-1} for argon with 13 m^3 h^{-1} for the primary pump. In stage 4 an air-cooled Pfeiffer turbo pump TMU521 was used, backed by a Varian Triscroll 300, giving a pumping speed of 500 L s^{-1} for argon.

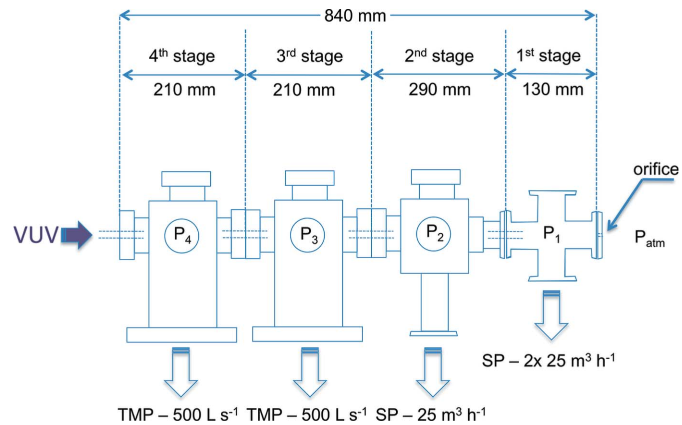

Figure 1
Schematic of the differential pumping system.

Whatever the flow regime, the system has to be designed with the smallest aperture holes. This is especially critical for the first orifice, which is in the viscous flow regime and for which the conductance depends on the square of the orifice diameter (Tamenori, 2010). This could be achieved by focusing the beam in both planes at this particular position. Nevertheless, the beam has to be focused vertically on the monochromator exit slits. Therefore, the only available degree of freedom was to match the entrance orifice with the horizontal focal plane. As a result, the horizontal and vertical focal plane are separated by 1090 mm. The optical design of the DISCO beamline has been described previously (Giuliani *et al.*, 2009). Briefly, the APEX branch uses a plane 250 lines mm^{-1} varied-line-spacing grating (GB) working in constant deviation mode (vertical deviation) with a spherical M6B mirror (R = 35.4 m curvature) of the beamline. The M6B mirror contributes to the beam vertical focusing onto the exit slits (selection of the wavelength), as well as the varied line spacing of the grating. At 3346 mm downstream from the grating the beam is deflected horizontally by a cylindrical mirror (M7, R = 37.9 m curvature) onto the exit slits located 5000 mm downstream. M7 controls the horizontal focusing of the beam. The deviation angles are grazing (GB, M6B: 15°; M7: 11°) in order to reach the spectral range down to 60 nm.

3. Mechanical aspect

The top panel in Fig. 2 shows the beam size as calculated by ray tracings of 90% of the rays. The differential pumping assembly is composed of four vacuum chambers separated by conductance-limiting sections. The conductance-limiting elements have been designed taking into account the theoretical size of the beam to which 10% was added. With a beam divergence of 16 mrad horizontally and 5 mrad vertically a spot size of 1.6 mm (horizontal) by 2.33 mm (vertical) is obtained for the horizontal focal plane placed at 1090 mm from the monochromator exit slits (see Fig. 2, top panel). The orifice is placed at this particular position. The first stage is separated from the atmosphere by an aperture hole of diameter 1 mm mounted on a DN40KF six-way cross. An

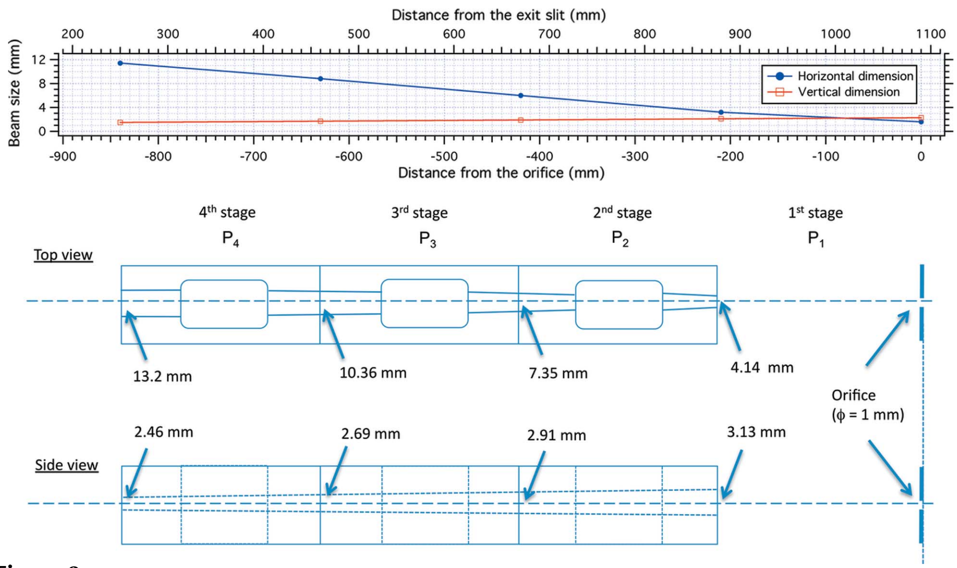

Figure 2
Theoretical beam size from ray tracing at 90% of the rays (top panel) and inner body and orifice aperture (bottom panel) to limit gas conductance.

inner body, inserted inside the chambers, was machined by wire cutting to adapt to the beam size in both vertical and horizontal directions, as seen in Fig. 2.

For the inner body, the general tolerances on linear dimensions are ± 0.05 mm for dimensions below 6 mm and ± 0.1 mm for dimensions below 30 mm. Geometrical tolerances of parallelism and perpendicularity are ± 0.02 mm with regard to the machining reference. It was mandatory to avoid the inner body which may take a 'banana' shape. Hence, rectitude on the beam axis of 50 µm was achieved. In order to obtain a perfect alignment of the four successive stages the whole system was placed on wedged blocks levelers. Pressure screws ensured translations along the horizontal plane. Taylor–Hobson reference spheres were used to align the system on the theoretical beam. Vacuum chambers and the innerbody were fabricated by the Rial Vacuum Company (Italy).

4. Performance tests

Vacuum measurements were made using capacitance gauge Oerlikron CTR 91 (high-pressure stage 1 and 2) and combined Pirani–Cold cathode gauges ACC1009 from Alcatel. The system was tested with argon and neon. The apparent pressure in stages 3 and 4 (ion gauges) had to be corrected using the appropriate factors from the gauges technical sheet.

The differential pumping system is separated from the beamline by a gate valve. After complete stabilization of the system, which may take up to a few minutes, the valve was opened. Fig. 3 compares the pressure measured over the whole beamline range before and after opening the differential pumping system.

It appears that the vacuum is degraded only for the elements closest to the differential pumping. The vacuum at the front end is not affected by the use of differential pumping. Furthermore, monitoring residual gas analyzers in the front end did not show any increase in argon and neon partial pressure after 2 h of continuous functioning (data not shown). Thus, the differential pumping system does not affect the quality of the vacuum inside the storage ring. In addition, the dipole is 14 m upstream from the gamma trap; the front end is protected by a delay line, one fast valve and three regular valves, ensuring perfect security for the ring vacuum in case of failure (Giuliani *et al.*, 2009). The gate valve, which allows isolation of the system from the beamline, is interlocked to the turbo pump speed and state.

Nevertheless, the pressure at several optical elements, such as the monochromator and M7 mirror, is notably increased. To avoid carbon contamination on these optical elements, the use of high-purity gases, such as Alpha Gas 2 from Air Liquide, with minimum carbon-containing species is mandatory.

5. Photon flux measurements in both gases

For transmission measurements, a Physikalisch-Technische Bundesanstalt (Germany) calibrated AXUV 100 photodiode (International Radiation Detector, USA) was mounted on a T-shaped vacuum pipe placed on the first stage. Neon, argon and nitrogen were admitted on the other port of the 'T' through a valve. The I_0 curve was obtained with the gas valve closed, giving a pressure better than 10^{-1} Pa in the first stage.

These measurements are compared with the transmission of atmospheric air and to the valve MgF_2 window, as shown in

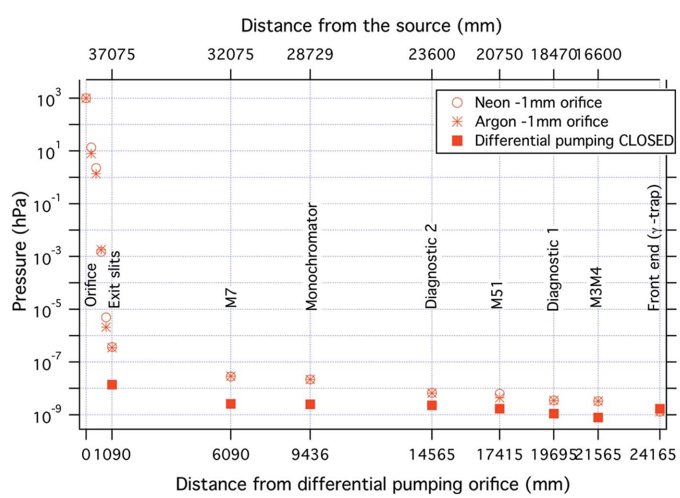

Figure 3
Pressure measured at different parts of the beamline for neon (open symbols) and argon (asterisk). Full squares refer to measurements for which the differential pumping had been isolated from the beamline.

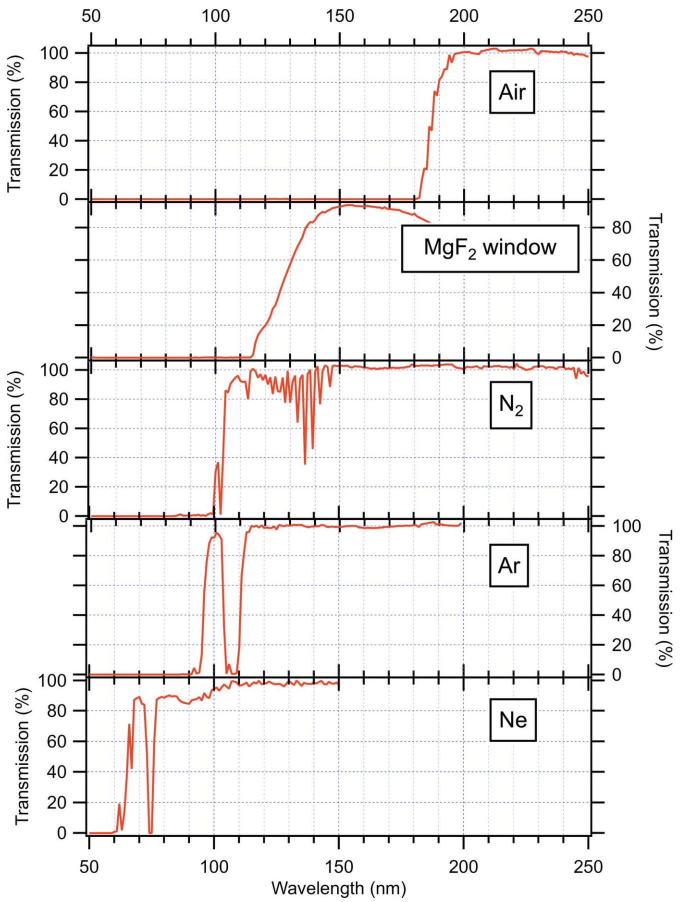

Figure 4
Transmission of the differential pumping recorded in the 250–50 nm range in one atmosphere (1013 hPa) of air, nitrogen, argon and neon with the differential pumping in operation for 400 mA ring current and top-up machine mode and 2000 µm slits. A gate valve bearing a MgF_2 window separates the beamline from the differential pumping.

Fig. 4. Below 114 nm the MgF_2 transmission is close to zero. The advantage of the differential pumping system is obvious: photons below 114 nm down to 60 are delivered at atmospheric pressure for the first time.

An additional benefit of the system is that it may also be used to suppress higher-order light from the monochromator, thus acting as a gas filter (Mercier *et al.*, 2000).

6. Conclusion

We report the performance of a differential pumping system, which is placed on the atmospheric pressure experience (APEX) branch of the DISCO beamline at the SOLEIL synchrotron radiation facility (Gif-sur-Yvette, France). This system enables the delivery of VUV photons (down to 60 nm) at atmospheric pressure in rare gases atmosphere. Its impact on the beamline vacuum is moderate and it has no incidence on the front-end vacuum level and quality. It may thus be used continuously without any perturbation on the storage ring or the beamline. The system allows photons to be transmitted in the 114–60 nm range, which is below the cut-off of MgF_2 windows.

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References

Chan, W. F., Cooper, G. & Brion, C. E. (1991). *Phys. Rev. A*, **44**, 186–204.
Chan, W. F., Cooper, G., Guo, X. & Brion, C. E. (1992). *Phys. Rev. A*, **45**, 1420–1433.
Chan, W. F., Cooper, G., Guo, X., Burton, G. R. & Brion, C. E. (1992). *Phys. Rev. A*, **46**, 149–171.
Giuliani, A. *et al.* (2009). *J. Synchrotron Rad.* **16**, 835–841.
Gog, T., Casa, D. M., Kuzmenko, I., Krakora, R. J. & Bolin, T. B. (2007). *J. Synchrotron Rad.* **14**, 339–344.
Hershcovitch, A. (1998). *Rev. Sci. Instrum.* **69**, 868–873.
Johnson, C. W. (1988). *Ann. Rev. Biophys. Chem.* **17**, 145–166.
Ladenburg, R. & Van Voorhis, C. C. (1933). *Phys. Rev.* **43**, 315–321.
Mercier, B., Compin, M., Prevost, C., Bellec, G., Thissen, R., Dutuit, O. & Nahon, L. (2000). *J. Vac. Sci. Technol. A*, **18**, 2533–2541.
Nebiki, T., Kabir Hasnat, M. & Narusawa, T. (2006). *Nucl. Instrum. Methods Phys. Res. B*, **249**, 226–229.
O'Hanlon, J. F. (2003). *A User's Guide to Vacuum Technology*. New York: John Wiley and Sons.
Pinkoski, B. T., Zacharia, I., Hershcovitch, A., Johnson, E. D. & Siddons, D. P. (2001). *Rev. Sci. Instrum.* **72**, 1677–1679.
Rozanov, L. N. & Hablanian, M. H. (2002). *Vacuum Technique*. New York: Taylor and Francis.
Schumann, V. (1903). *Smithson. Contrib. Knowl.* **49**, 1413.
Tamenori, Y. (2010). *J. Synchrotron Rad.* **17**, 243–249.
Watanabe, K., Inn, E. C. Y. & Zelikoff, M. (1953). *J. Chem. Phys.* **21**, 1026–1030.