

## Development of a Differential Pumping System of Soft X-Ray Beamline for Windowless Experiments under Normal Atmospheric Conditions

In synchrotron radiation facilities, thin metal and polymer films are usually used as a vacuum window to separate the ultra-high vacuum of the storage ring or the optics chamber from the high-pressure environment of the experimental apparatus. A beryllium (Be) window is often used as a standard vacuum window owing to its high optical transmission of the X-ray beam. However, Be windows absorb low energy photons, and cannot be used for a soft X-ray beamline. Instead, the standard procedure in a soft X-ray beamline is to install all the equipment necessary for experiments inside the high-vacuum chamber. This method can have drawbacks as well. On one hand, the high-vacuum chamber produces an ideally clean environment. On the other hand, technologically significant phenomena take place under higher-pressure regions (1~760 mbar). Recently, the demands to study samples under a high-pressure environment have been increasing in the soft X-ray region. For example, several groups have attempted to obtain photoemission data at pressures of up to 100 mbar by separating the sample region from the beamline with a vacuum window [1]. The vacuum windows used in the soft X-ray regions need to be ultra-thin to transmit the photons to the sample region. However, thin films are fragile and have difficulty withstanding a high pressure difference. Furthermore, organic contamination of the vacuum window reduces the photon intensity in the absorption edges of the light elements. In order to overcome these complications, the development of a windowless method must be investigated. A windowless connection between a

high-vacuum beamline and a high-pressure sample chamber would provide an opportunity that opens new scientific possibilities in soft X-ray sciences.

One approach to overcoming the window problem is to use a differential pump [2]. In a differential pump, the conductance between two vacuum chambers is limited by an open aperture or pipes. It allows photons of all energy ranges to pass freely along a line of the light path between the two different pressure regions. A challenge of the present investigation is to create a windowless connection between an experimental chamber filled by 1 atm helium (He) and the high-vacuum beamline. Since soft X-rays below 1.5 keV are fairly absorbed in atmospheric air, a He path was used instead of an air path. While He gas has a higher transmission than air in the soft X-ray region owing to its smaller atomic number, it is more difficult to evacuate He using vacuum pumps. In this study, a differential pumping system was developed for use with windowless soft X-ray experiments under normal atmospheric conditions. The performance of the differential pump was evaluated by connecting it to both atmospheric air and helium environments. The transmission of soft X-rays was measured under the condition of atmospheric helium to demonstrate the performance of the apparatus.

Figure 1 shows a photograph of the present differential pumping system. The details of the system were described in a recent report [3]. The apparatus consists of an aperture-based four-stage differential pump, which was designed on the basis of a simple

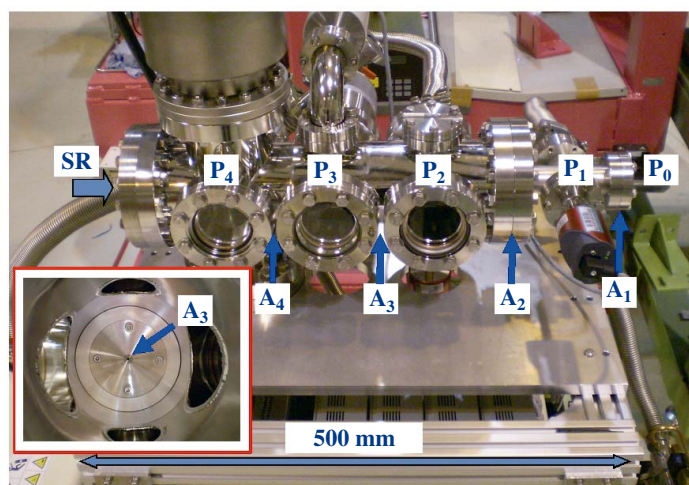


Fig. 1. Photograph of the fabricated differential pumping system.  $A_x$  ( $x = 0 - 4$ ) is the location of each conductance-limiting aperture, and  $P_x$  ( $x = 0 - 4$ ) is the pressure at each differential pumping stage. Inset is a photograph of the aperture installed between the third and fourth stages at BL27SU.

model calculation. The design has relatively short conductance-limiting components that allow easy installation and alignment of the system on the synchrotron beamline. The total length of the system is about 500 mm, which is significantly shorter than those of other similar systems previously reported [2].

Figure 2 shows the pressure distribution curves obtained from the simulation and the test measurement of the system. The pressure  $P_0$  in the sample region is  $1 \times 10^5$  Pa (= 1 atm). When  $P_0$  is the atmospheric air, the pressure at the uppermost stage ( $P^4$ ) is  $8.5 \times 10^{-5}$  Pa. The present apparatus achieved differential pressures of about 9 orders of magnitude less than the atmospheric pressure of air. The next challenge was to connect the experimental chamber filled with 1 atm of helium gas to the high-vacuum beamline in the windowless system. The pressure of the  $P_4$  section increased by a factor of about 40, compared with the  $P_4$  value for air. The pressure deteriorated for mainly two reasons [3]. The first reason is that a gas mass increases the gas flow, as was the case with helium. The second reason is that the turbomolecular pumps had lower pumping speeds with helium than with air, and the lower speeds reduced the pumps' performance. Although the performance of the system degraded by about one order of magnitude when using helium, the observed  $P_4$  value ( $3.3 \times 10^{-3}$  Pa) is sufficient to connect this system to the beamline. The present system provides excellent isolation between the atmospheric pressure of the sample environment and the high-vacuum of the beamline across the short distance.

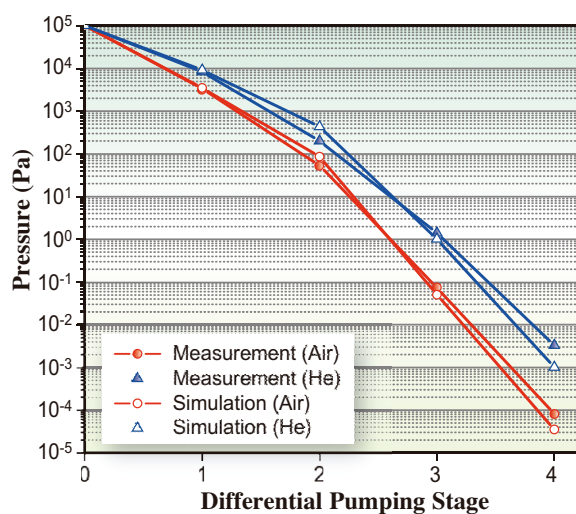


Fig. 2. Performance measurement of the present differential pumping system. The pressure distribution curves were measured for the atmospheric pressures of air (solid circles) and helium (solid triangles). The calculated pressure for the optimized design is also indicated for air (empty circles) and helium (empty triangles).

Figure 3 shows the transmission curves of soft X-rays under atmospheric helium. The experimentally determined transmission curves are nearly identical to the simulated ones. The transmission at 550 eV (the oxygen *K*-edge region) measured at 10 mm downstream of the first aperture is about 85%. When the photon flight path in the atmospheric pressure region is expanded to 50 mm, more than 70% of the incident photons are transmitted to the detector. Although the transmission is decreased in the low photon energy region, about 60% of the incident photons are transmitted to the detector at 350 eV. The fabricated differential pump was a success: low energy soft X-ray photons passed through the atmospheric pressure environment.

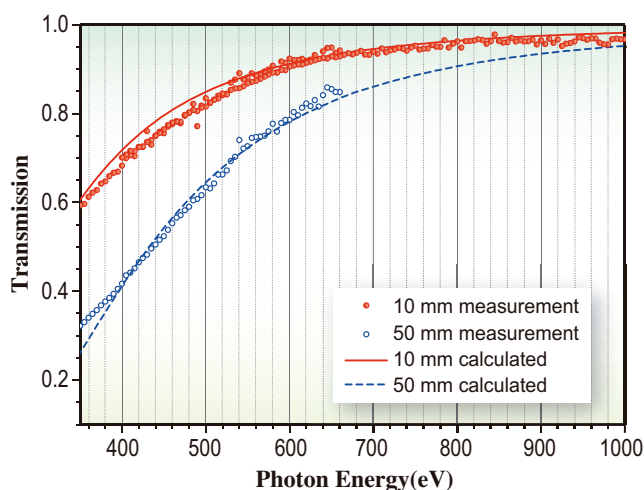


Fig. 3. Experimental and calculated transmission curves of soft X-ray photons obtained under helium-path conditions. The experimental transmission curves were measured at the helium-path lengths of 10 mm (solid circles) and 50 mm (empty circles). The solid (10 mm) and dashed (50 mm) lines indicate the simulated transmission curves of the fabricated differential pumping system.

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## References

- [1] D.F. Ogletree *et al.*: Rev. Sci. Instrum. **73** (2002) 3872.
- [2] T.Gog *et al.*: J. Synchrotron Rad. **14** (2007) 339.
- [3] Y. Tamenori: J. Synchrotron Rad. **17** (2010) 243.