



MEDSI 2014, Oct. 21, 2014

***In situ* removal of carbon contamination from optics, suppression of higher harmonics in the carbon *K*-edge region, and development of low-cost and high-performance non-evaporable getter (NEG) pumps**

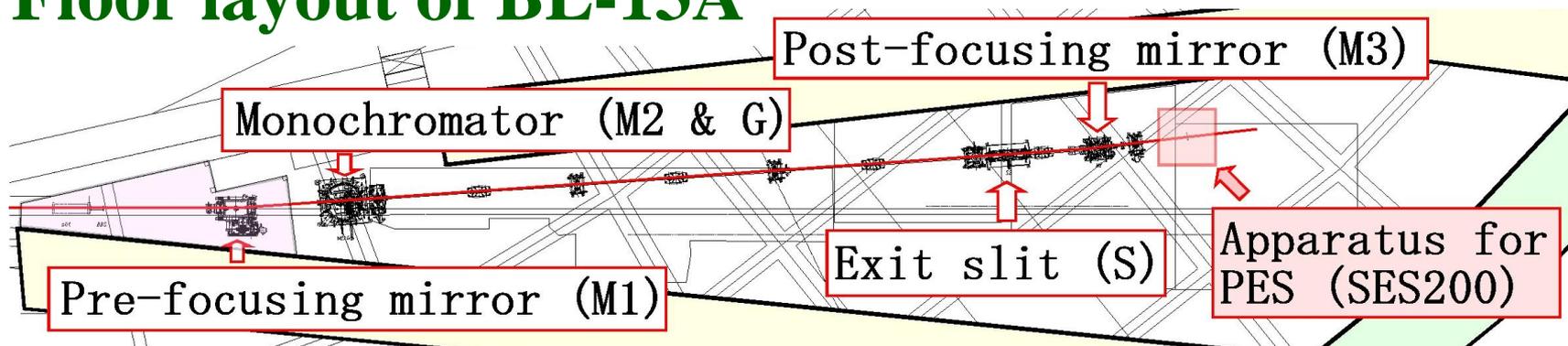
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Photon Factory,  
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# 1. *In situ* removal of carbon contamination from optics using SR-activated oxygen

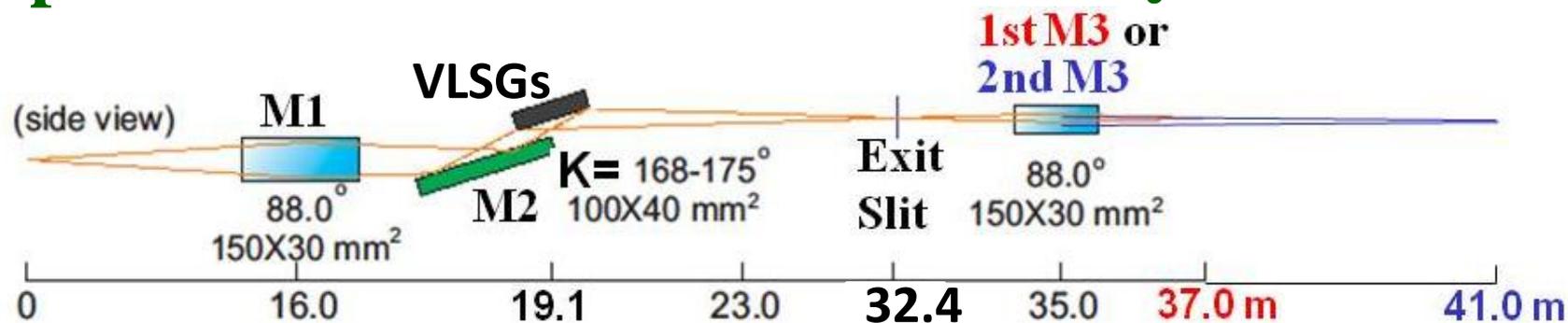
## 1-1. Undulator-based VSX beamline, BL-13A at PF

### Floor layout of BL-13A



### Optics of BL-13A

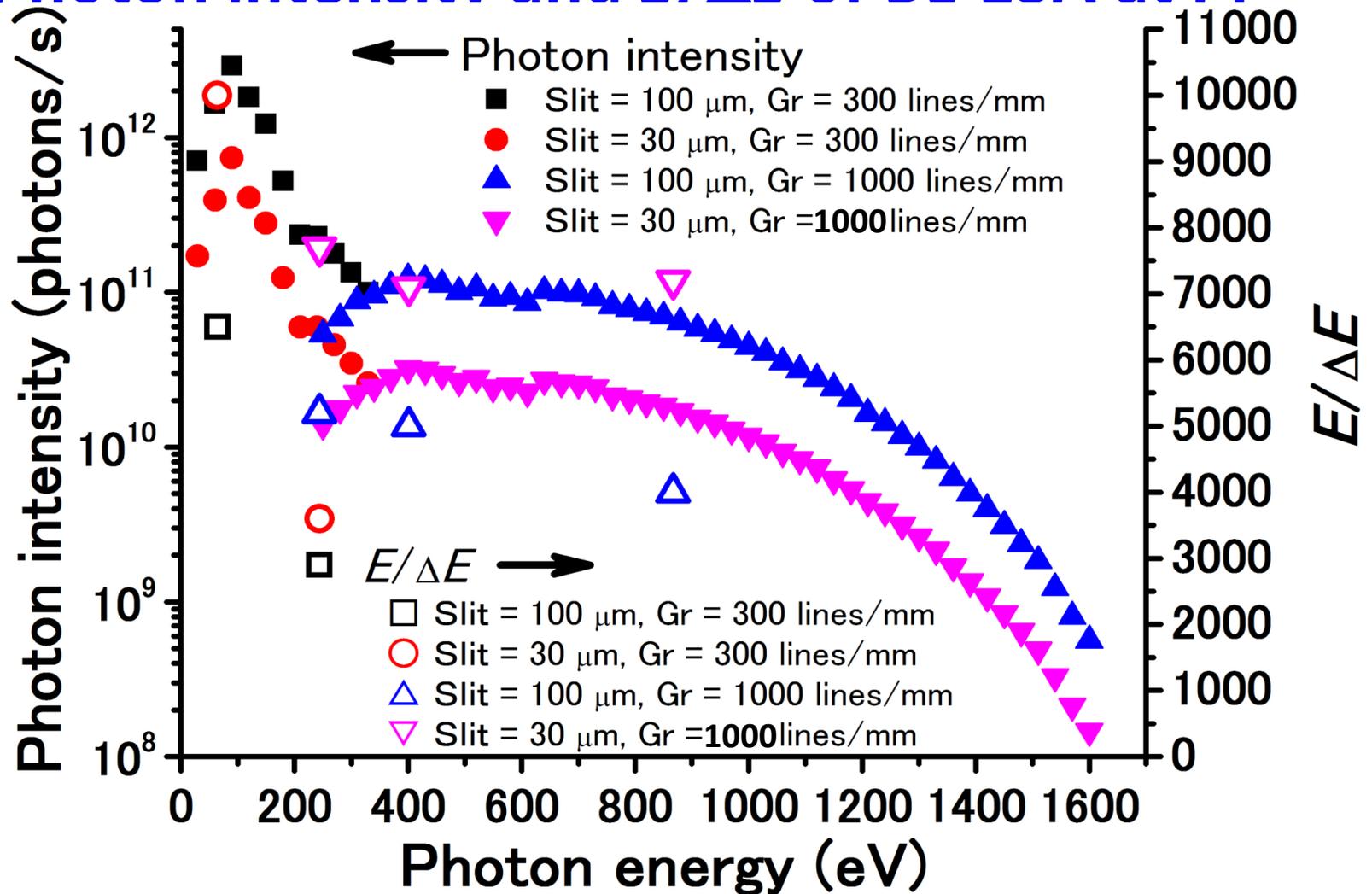
Constructed by TOYAMA



★ Base pressure of BL-13A is less than  $1 \times 10^{-8}$  Pa

[A. Toyoshima *et al.*, J. Vac. Soc. Jpn. 54, 580 (2011).]

# 1-2. Photon intensity and $E/\Delta E$ of BL-13A at PF



$h\nu = 30\text{--}1,600$  eV; Photon flux,  $10^{12}\text{--}10^8$  photons  $\text{s}^{-1}$

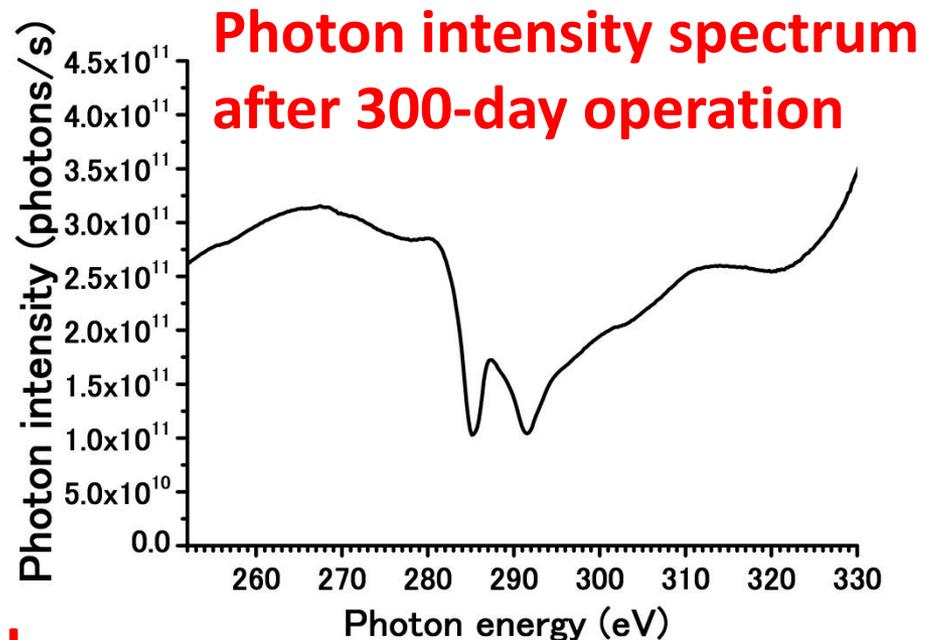
Photon-energy resolution ( $E/\Delta E$ ), 10,000 at  $h\nu = 64$  eV

[A. Toyoshima *et al.*, J. Phys.: Conf. Ser. **425** (2013) 152019.]

# 1-3 To achieve hydrocarbon-free UHV ( $<1 \times 10^{-8}$ Pa)

- ★ **The chambers** for optics and the metallic parts in the chambers (SS304 or Al), **were polished electronically and rinsed with pure water.** (TOYAMA)
- ★ **No lubricating oil was used** in the chamber.
- ★ The bearings were cleaned with organic solvent.
- ★ **Pumped with oil-free (TMP+FT+oil-sealed RP)+SIP+NEG.**
- ★ Baked at  $90^{\circ}\text{C}$  to avoid decomposition of viton.
- ★ However, optics are contaminated with carbon. Hydrocarbons adsorbed on optics and chambers are thought to be responsible.

⇒ ***In situ* method is required.**



# 1-4. Methods to remove carbon contamination

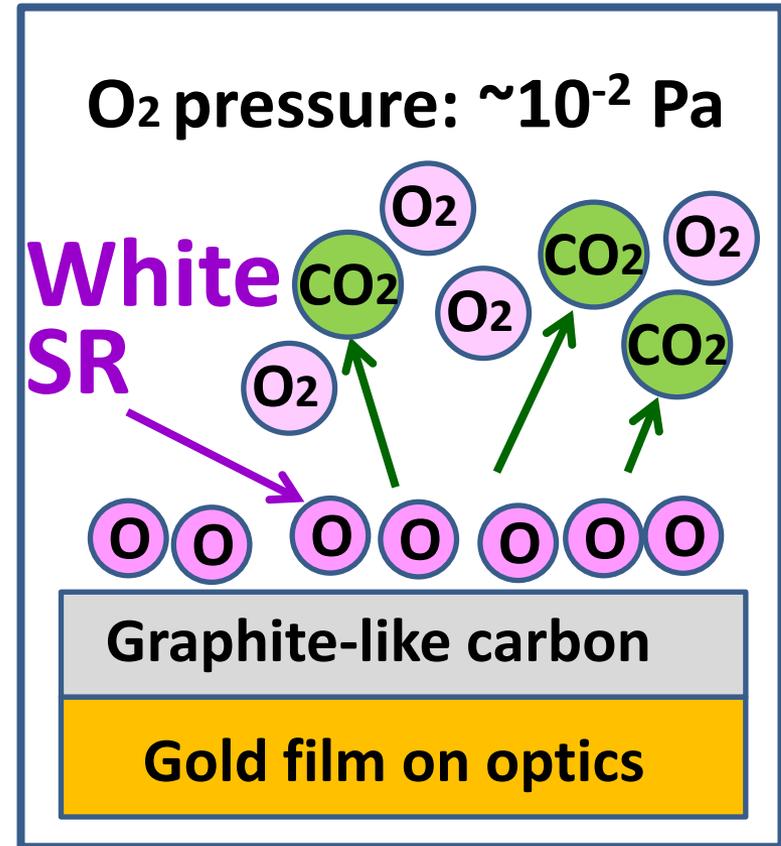
1. *In situ* cleaning using white-SR-activated oxygen [Warburton & Pianetta, 1992] [Hamamoto *et al.*, 2005].

2. *In situ* cleaning with oxygen activated by a 172-nm excimer lamp [Hamamoto *et al.*, 2005].

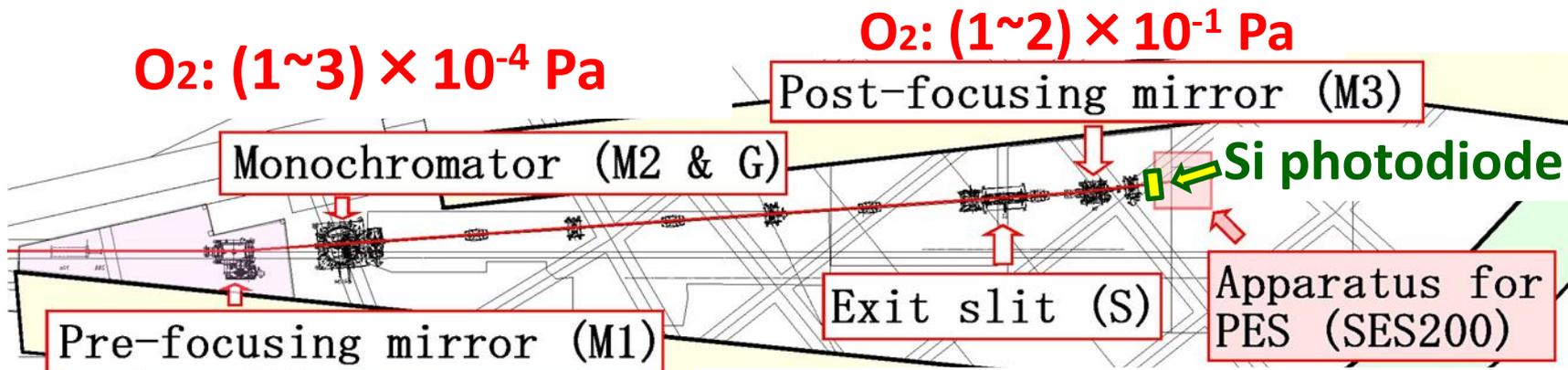
3. *In situ* dc oxygen discharge [Koide *et al.*, 1986; Koide *et al.*, 1987a; Koide *et al.*, 1987b; Koide *et al.*, 1988; Koide *et al.*, 1989).]

4. *In situ* plasma discharge in a mixture of oxygen and argon gases [Eggenstein *et al.*, 2001.]

5. Atmospheric-pressure ultraviolet (UV)/ozone cleaning [Harada *et al.*, 1991; Hansen *et al.*, 1993; Hansen *et al.*, 1994a; Hansen *et al.*, 1994b.]

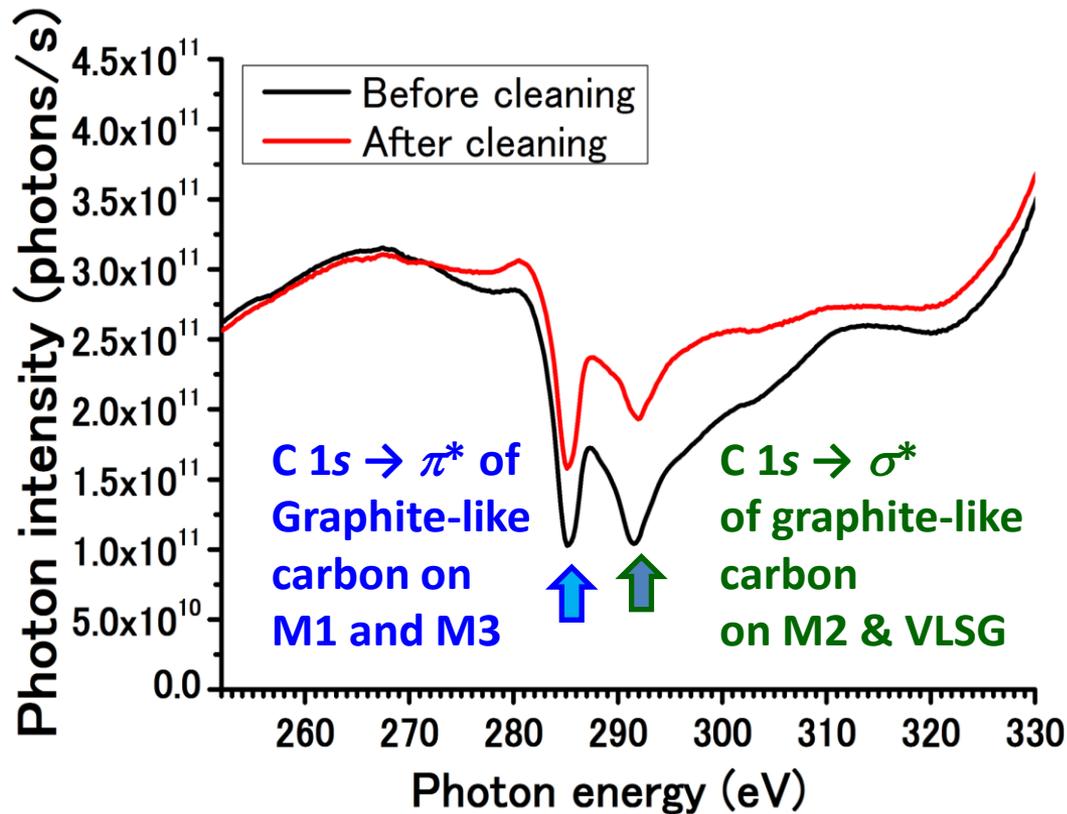


# 1-5. *In situ* carbon removal from optics of BL-13A (1)



$O_2: (1\sim3) \times 10^{-6} \text{ Pa}$

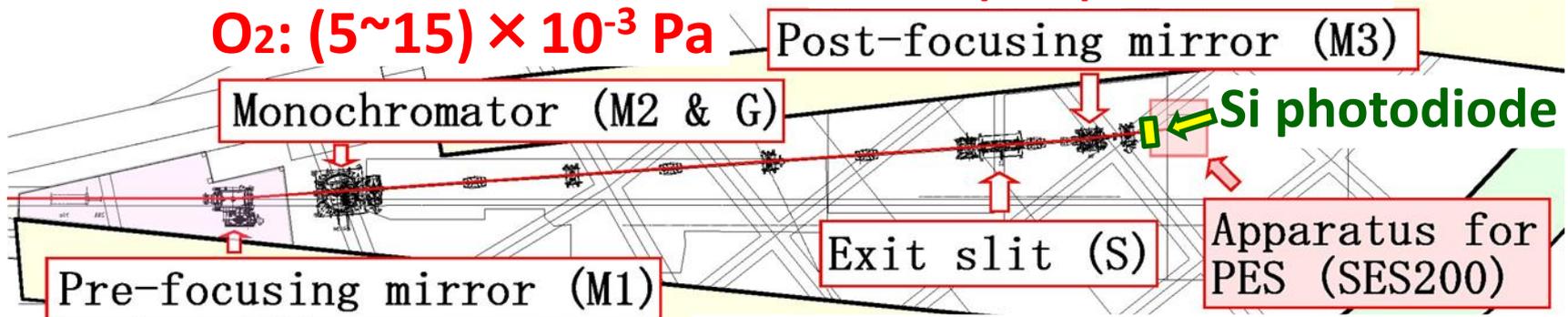
White SR beam exposure for 19 hours.



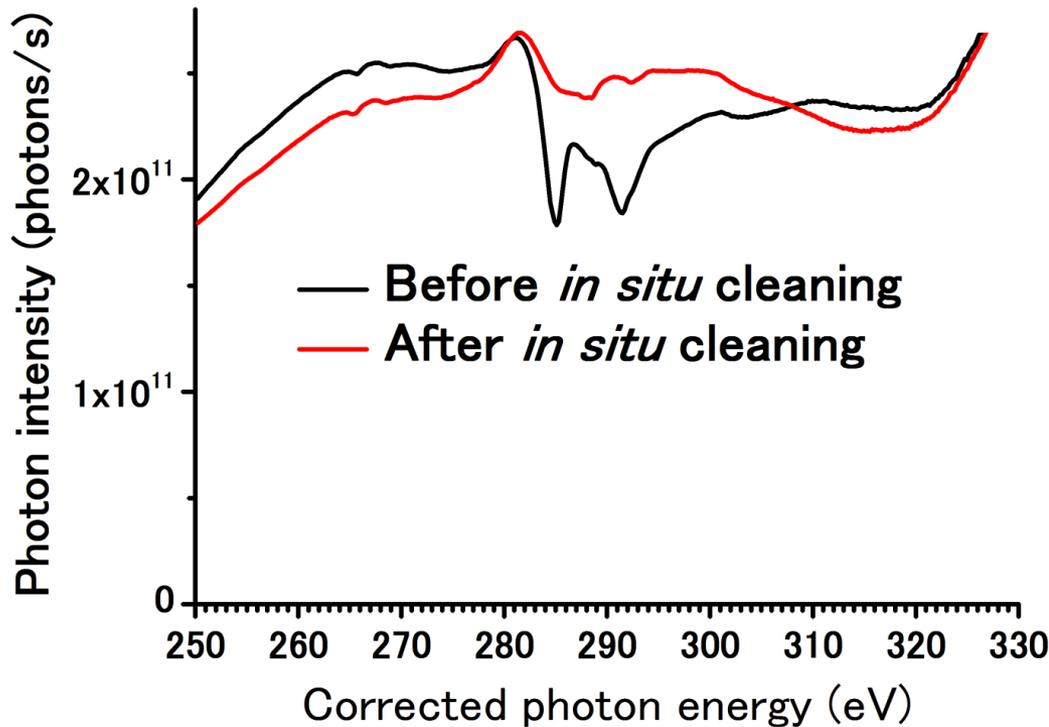
Carbon contamination of M3 was removed. Carbon contamination of M2 and VLSG was removed to some degree. The cleaning rate was estimated to be about 0.06 nm/h for M2 and the VLSG.

# 1-6. *In situ* carbon removal from optics of BL-13A (2)

$O_2: (1\sim4) \times 10^{-5} \text{ Pa}$



$O_2: (2\sim6) \times 10^{-4} \text{ Pa}$  White SR beam exposure for 20 hours.

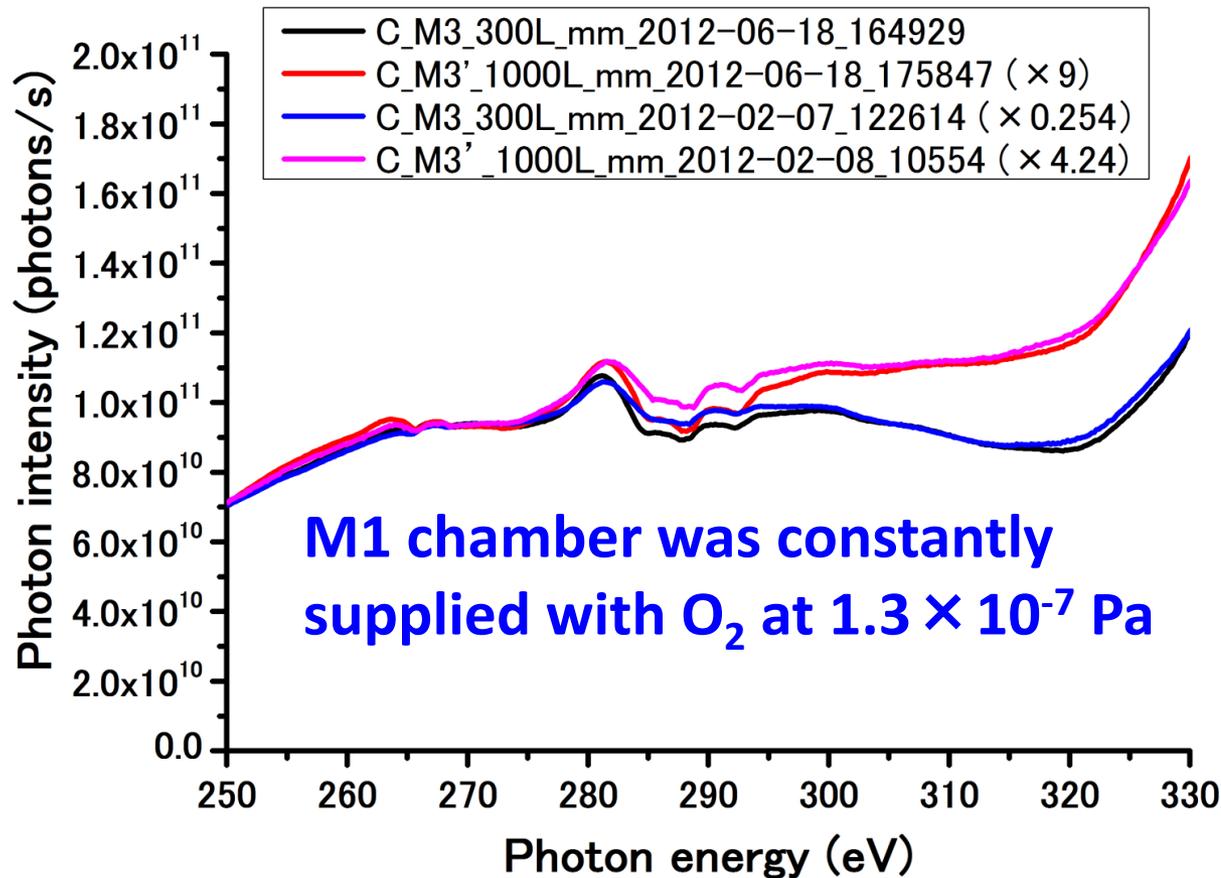


Carbon contamination of optics is almost removed. The remained dips (4% at 288.5 eV and 2% at 292.3 eV) are ascribed to the carbon buried in the gold film of the optics. The base pressure recovered to  $10^{-7}$ – $10^{-8}$  Pa in one day.

# 1-7. Preventing carbon contamination of optics

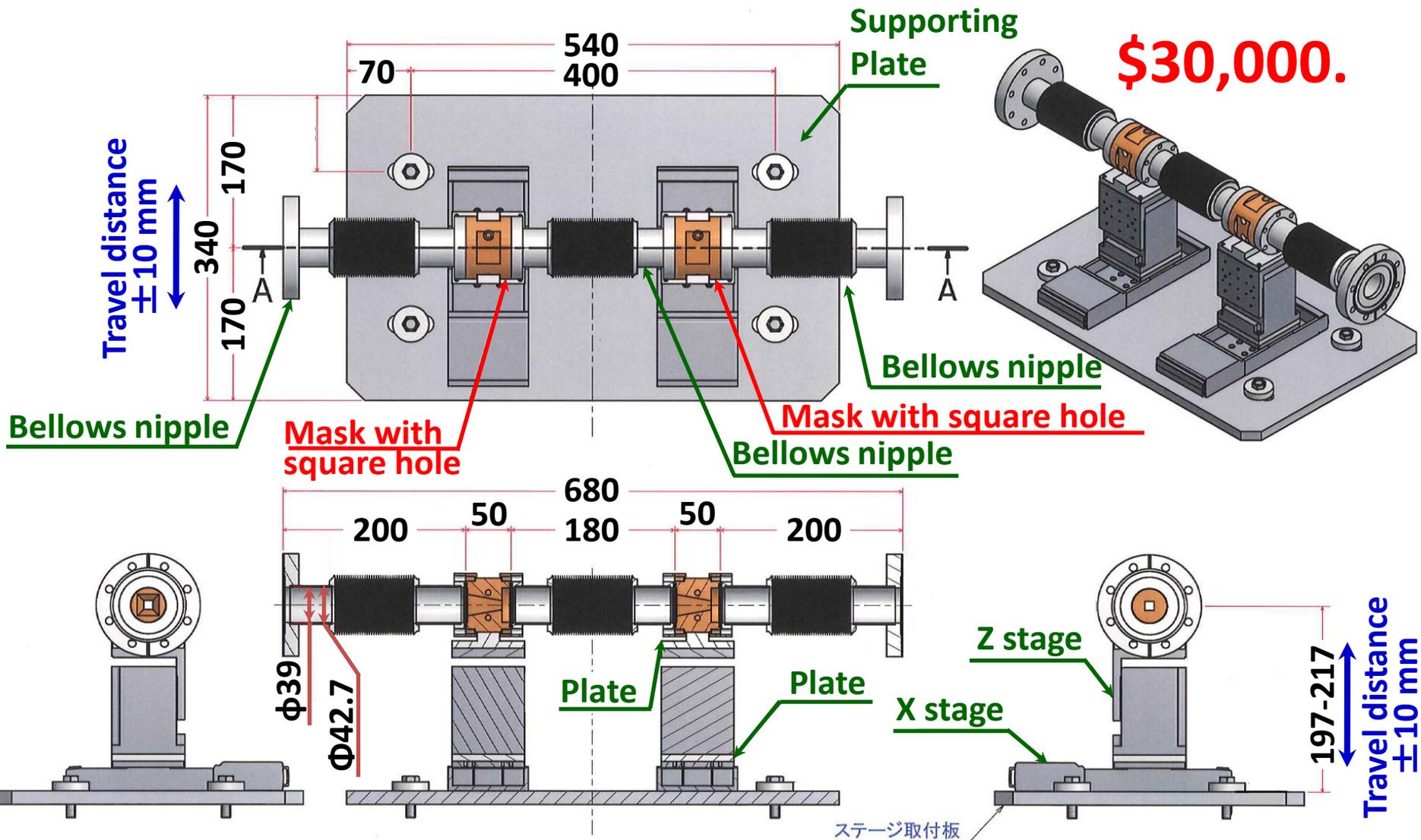
[W. K. Warburton & P. Pianetta, Nucl. Instrum. Methods Phys. Res. A **319** (1992) 240.]

Since optics are continuously irradiated by SR during beamline operation, **continuous exposure to oxygen gas with a pressure of  $10^{-5}\sim 10^{-7}$  Pa is effective to prevent carbon contamination.**



# 1-8. Quadruple water-cooling masks in BL-13

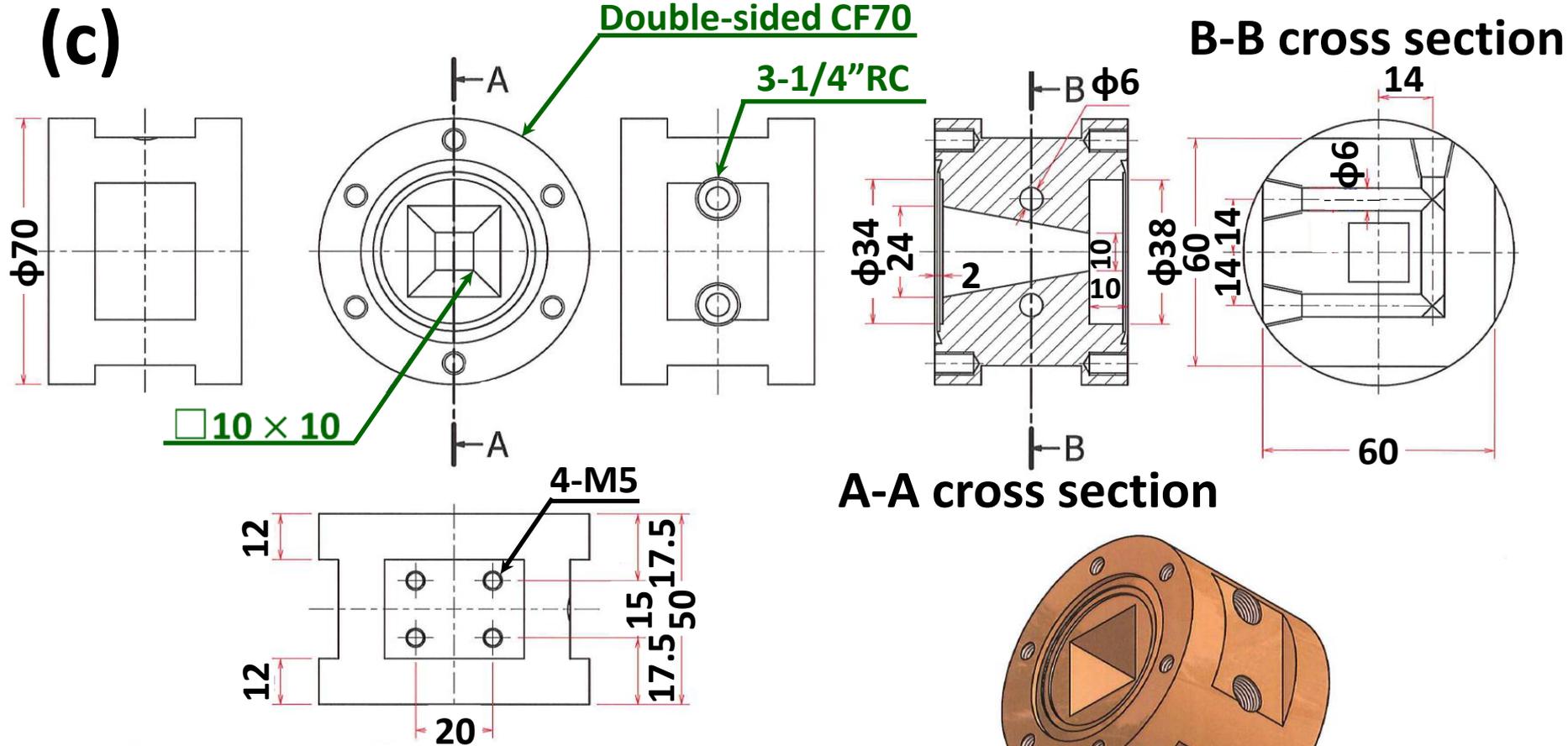
Low-conductance and low-cost masks for differential pumping.



A-A cross section

[K. Mase, et al., J. Vac. Soc. Jpn. 53, 454 (2010).]

# 1-9. One-Body Type 0.2% Be-Cu Water-Cooling Mask

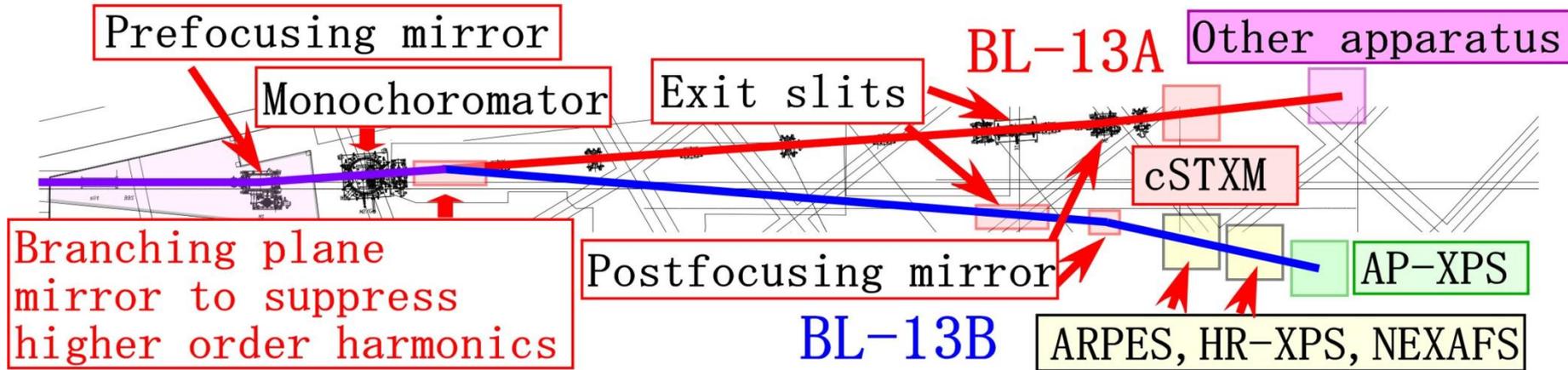


0.2% Be-Cu is ideal for masks because of High thermal conductivity ( $210 \text{ W m}^{-1} \text{ K}^{-1}$ ), hardness (HB 237), and low outgassing ( $5.6 \times 10^{-13} \text{ Pa (H}_2\text{) m s}^{-1}$ ).

[K. Mase, et al., J. Vac. Soc. Jpn. 53, 454 (2010).]

## 2. Suppression of higher order harmonics in the carbon *K*-edge region

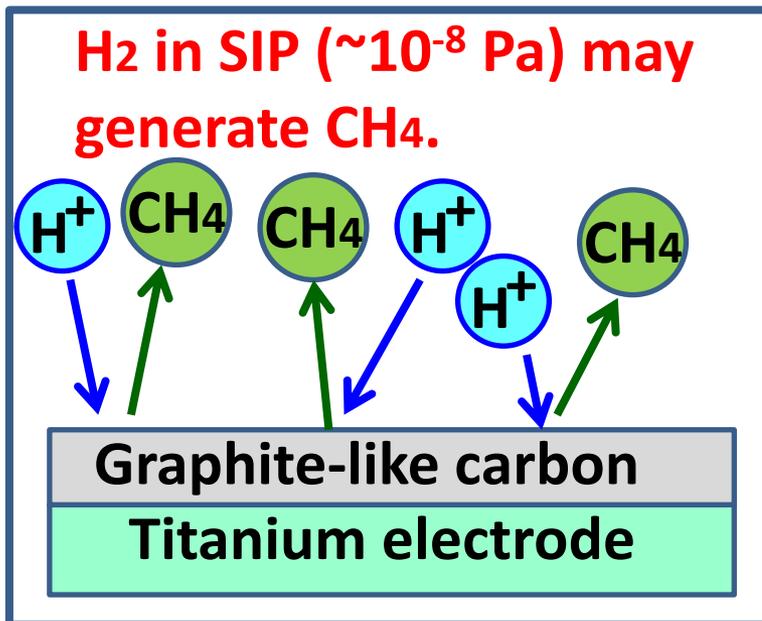
### 2-1. Undulator-based VSX branch beamline, BL-13B at PF



**BL-13B was constructed in March 2013 and was opened for users in Oct. 2013.**

### 3. Development of low-cost and high-performance non-evaporable getter (NEG) pumps

Sputter ion pump (SIP) may be the potential source of hydrocarbons when the electrodes are contaminated with carbon.

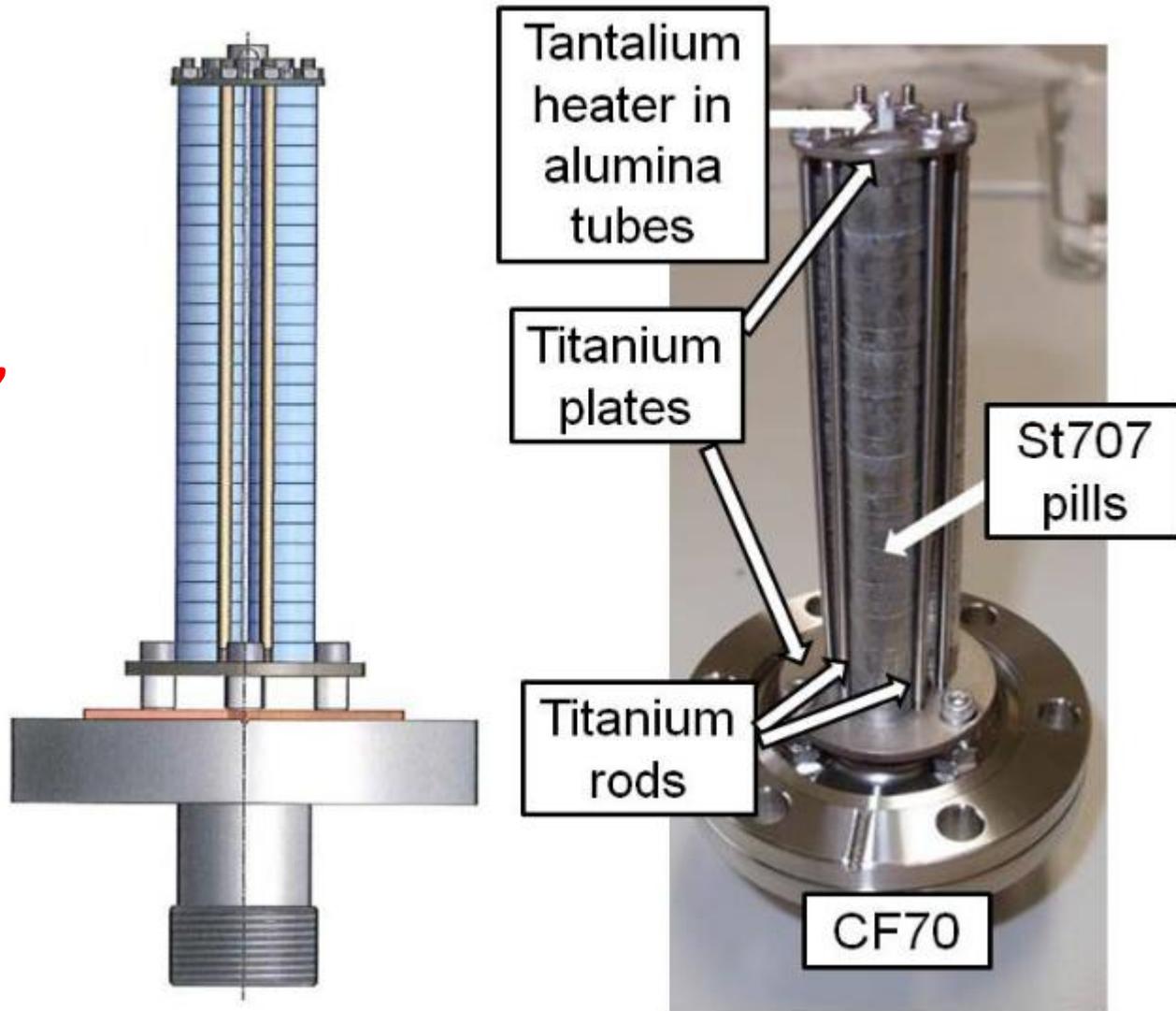


NEG pumps are ideal for VSX beamlines because of **high pumping speed for reactive gases, oil-free, compact, lightweight, evaporation-free, sputtering-free, sublimation-free, vibration-free, energy-saving, and low-cost.**

Disadvantages such as **short lifetime against frequent vent and no pumping speed for rare gases** do not matter in VSX beamlines.

### 3-1. Construction of NEG pumps

We developed a compact NEG pumps which consists of **St707 NEG pills ( $\phi 10 \times t3$ , 84 pieces)**, a Ta heater, and an flange with an electronic feedthrough. **NEG pills are activated at  $400^{\circ}\text{C}$  for 30 min.**





# Summary

1. Carbon contamination of all of the optics in a VSX undulator beamline is removed by **a *in situ* method using oxygen activated by non-monochromatized SR (white SR)**.
2. Higher-order harmonics in the Carbon *K*-edge region are suppressed by using **a Cr-coated mirror**. Carbon contamination of Cr mirror is removed using oxygen activated by white SR.
3. Low-cost and high-performance **non-evaporable getter (NEG) pumps are developed**.

## Acknowledgements

We are grateful to the staff of the PF, Mr. M. Tanaka, Mr. N. Ida, Mr. H. Kodama for their valuable support. Development of NEG pump is supported by Grant-in-Aid for Scientific Research 26390070.