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Zero-Length Conflat Fin-Type Nonevaporable Getter Pump Coated with Oxygen-Free Palladium/Titanium

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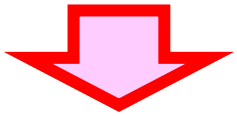
Profile: Leader of Vacuum System Team in charge of beamlines and endstations at the Photon Factory in KEK (KEK-PF), and also a beamline scientist in charge of three VSX beamlines (BL-13, 3B, and 11D) in KEK-PF. My mission is to develop new vacuum technologies for beamlines and endstations as well as to maintain vacuum systems using conventional vacuum components.

Introduction -What is the ideal vacuum pump in SR?-

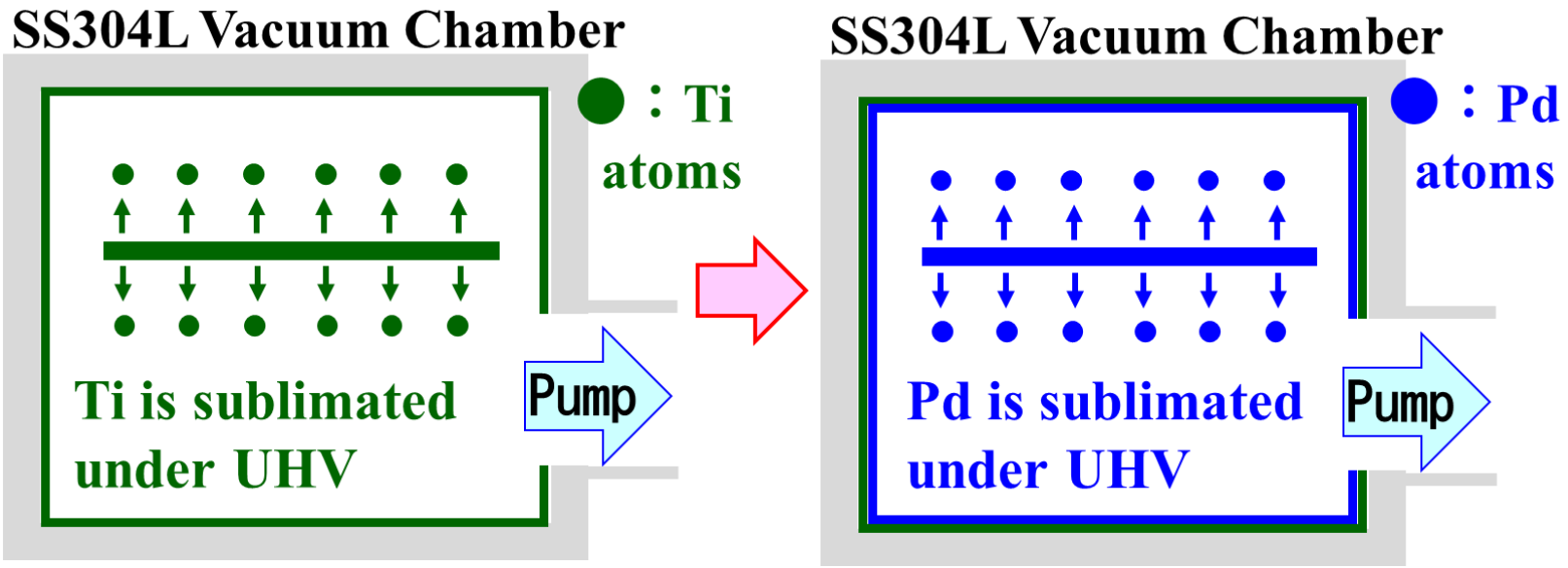
- ✓ In the initial pumping and baking oil-free vacuum pumps such as dry pumps (DPs) and turbomolecular pumps (TMPs) are required.
- ✓ To maintain ultra high vacuum (UHV) sputter ion pumps (SIPs) and/or nonevaporable getter (NEG) pumps are required.
- ✓ During user beamtime DPs and TMPs should be stopped to suppress vibration.
- ✓ Hydrocarbons in the chambers should be removed to suppress carbon contamination on the optics in the beamline.



- ✓ In order to meet all these requirements with low cost and low labor, we have developed a novel NEG named oxygen-free Pd/Ti. Oxygen-free Pd/Ti pumps residual H_2 and CO after baking at 133 ℃ for 12 h. In addition, due to the catalytic effect of Pd, the hydrocarbons in the chambers react with H_2O and/or O_2 during baking to form CO and CO_2, which are then pumped by TMP.

Oxygen-free Pd/Ti deposition

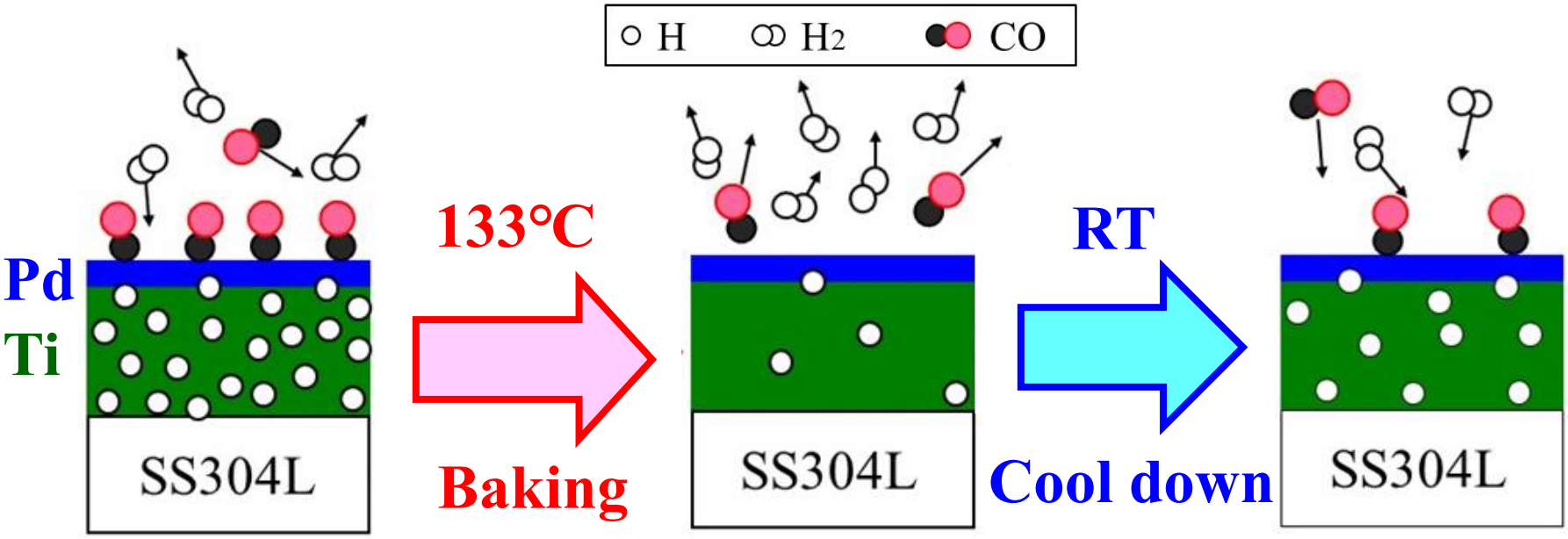
Pd/Ti thin films were deposited by sequential sublimation of Ti and Pd under UHV in range 10^{-7} to 10^{-8} Pa. This Pd/Ti was named oxygen-free Pd/Ti, because its oxygen content was estimated to be less than 0.05% .



International patent, PCT/JP2017/042682, Nov. 28, 2017.
Patents have been granted also in the EU, China, and Korea.
[T. Miyazawa *et al.*, J. Vac. Sci. Technol. A 36, 051601 (2018).]

Activation & pumping mechanisms of oxygen-free Pd/Ti

Since Pd surface has the property of dissociating H_2 into 2H at room temperature and diffusing H atoms in the Pd bulk, and chemisorbs CO at room temperature, oxygen-free Pd/Ti can evacuate H_2 and CO at room temperature after activation. Since Pd does not oxidize, the pumping performance does not be degraded even after repeated activation and exposure to air.



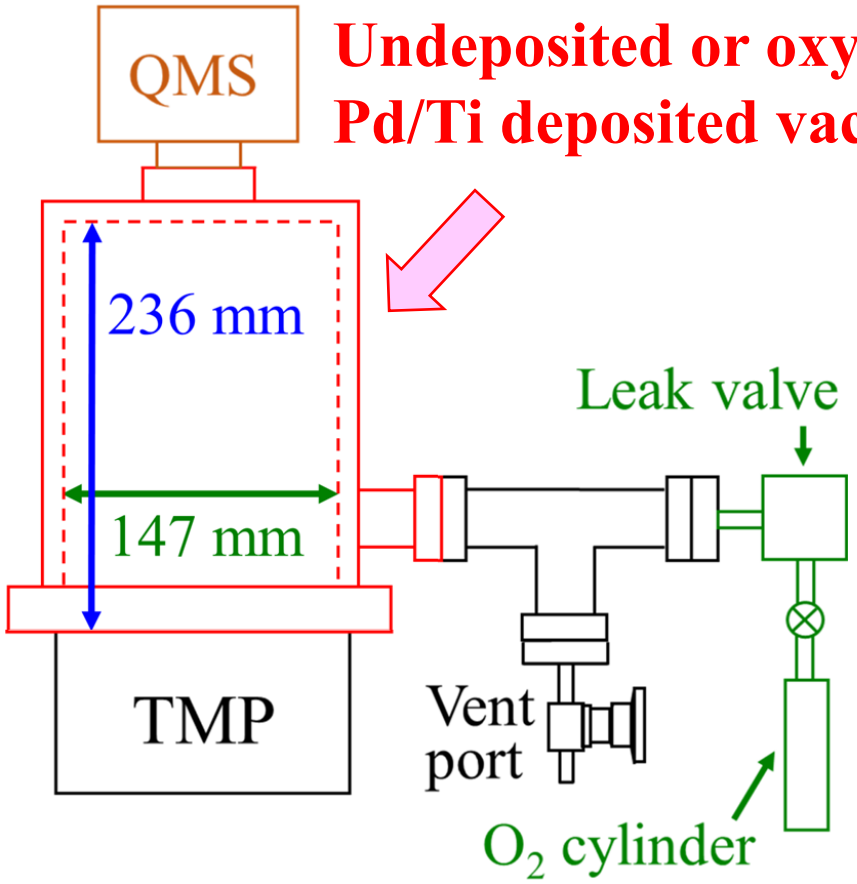
[T. Kikuchi *et al.*, AIP Conf. Proc. 2054 (2019) 060046].

Partial and total pressure measurements

Partial and total pressure curves of undeposited and oxygen-free Pd/Ti deposited vacuum vessels were measured with the apparatus shown below.

Prisma 80; Pfeiffer Vacuum

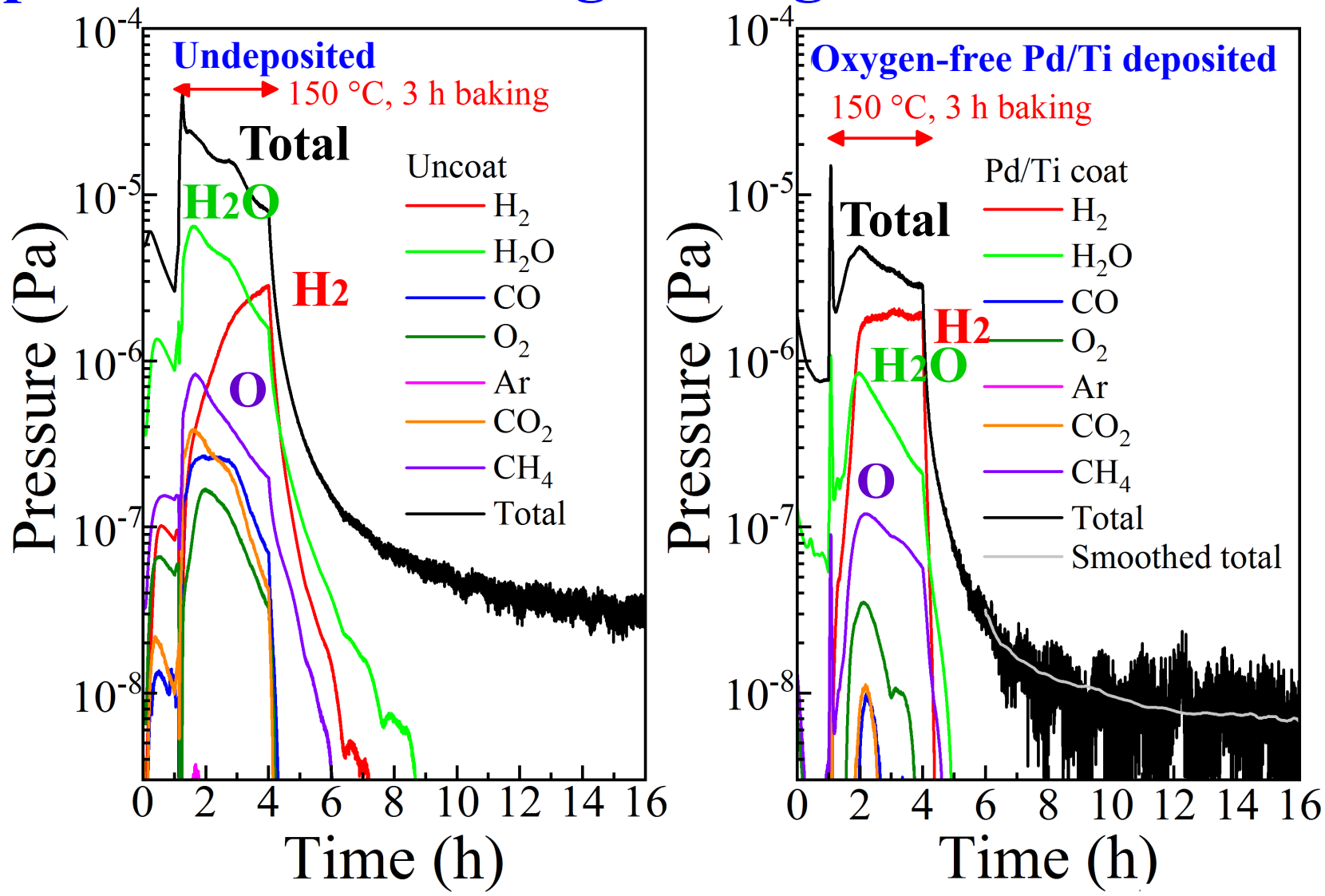
Total pressure was also measured with the QMS.



Pumpd by RP + FT + TMP

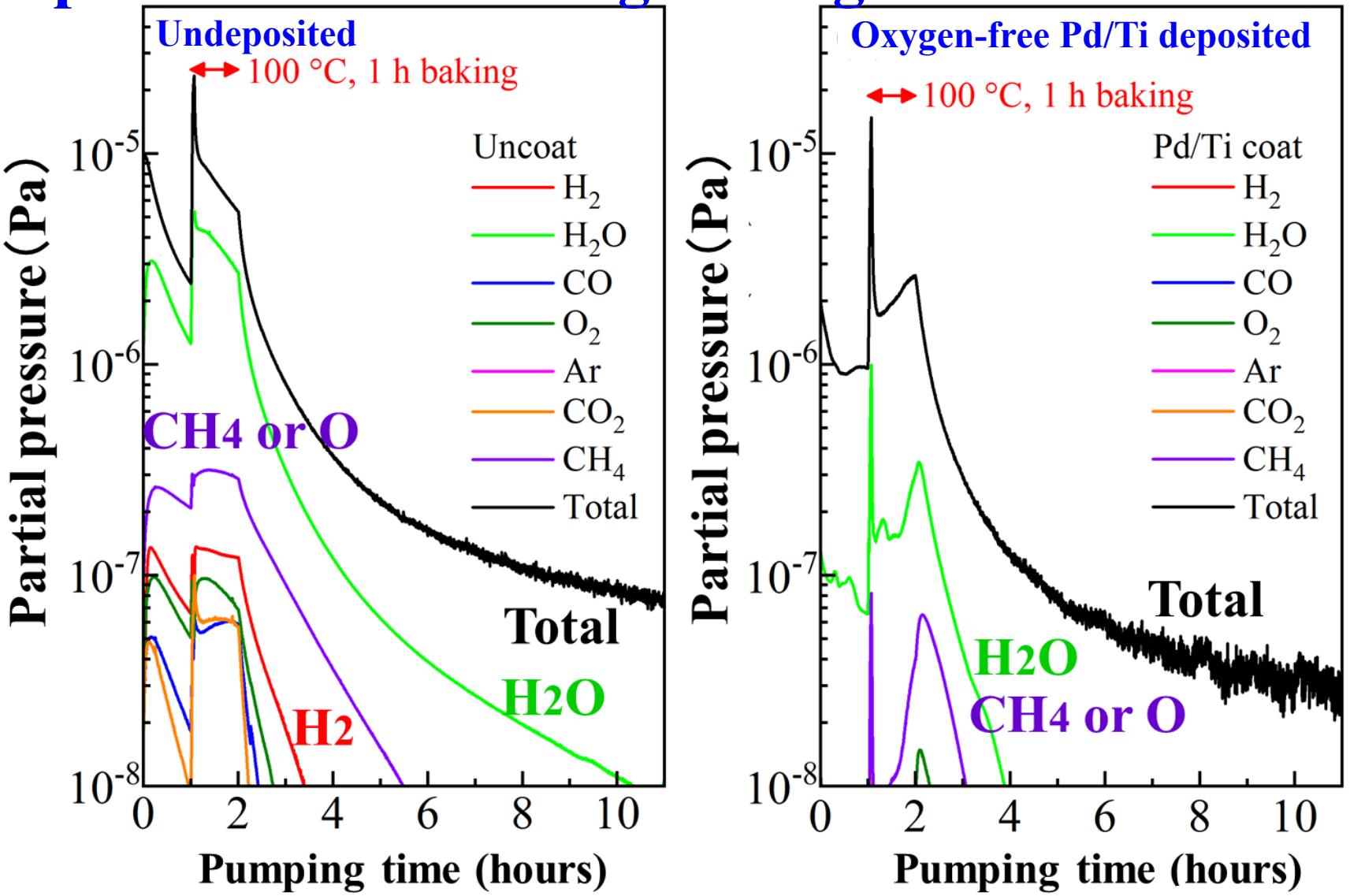
[T. Miyazawa *et al.*, to be published].

Pressure curves of uncoated and oxygen-free Pd/Ti deposited vessel during baking at 150 °C for 3 h



[T. Miyazawa *et al.*, to be published].

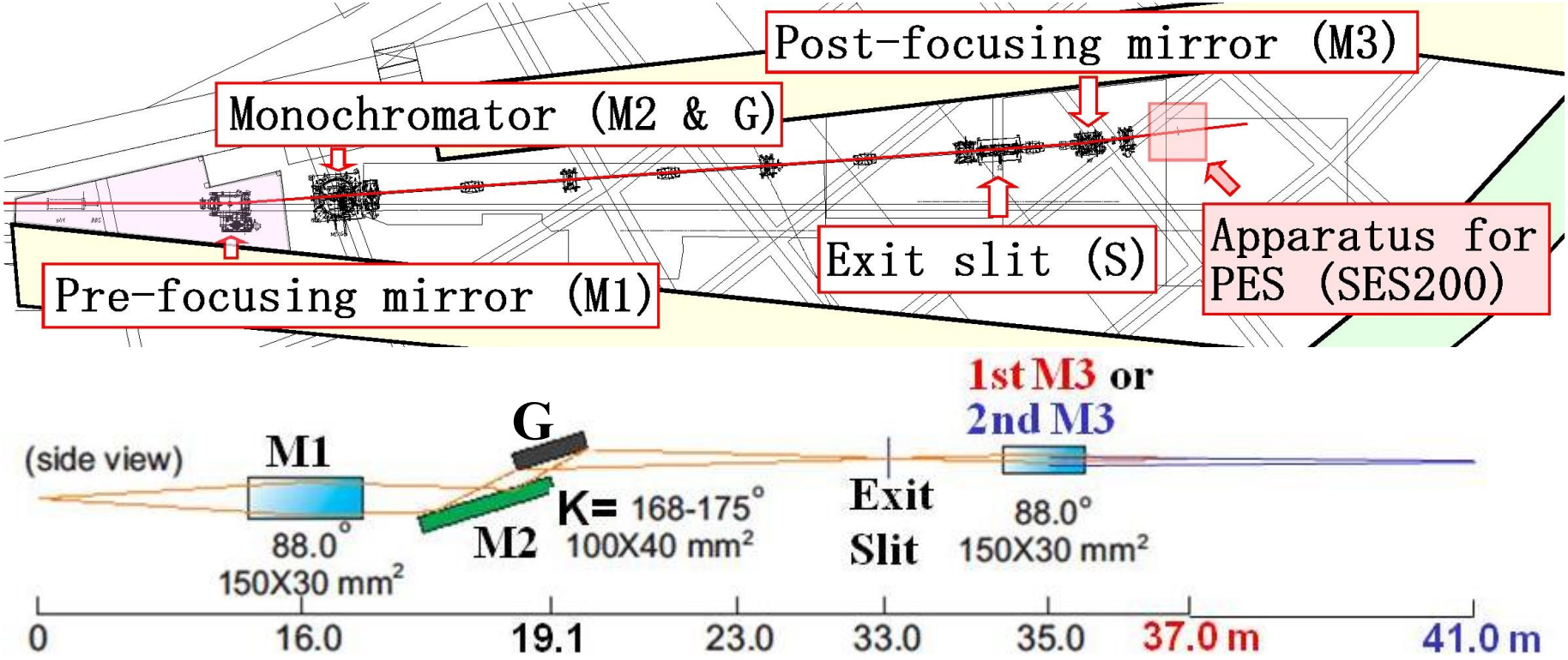
Pressure curves of uncoated and oxygen-free Pd/Ti deposited vessel during baking at 100 °C for 1 h



[T. Miyazawa *et al.*, to be published].

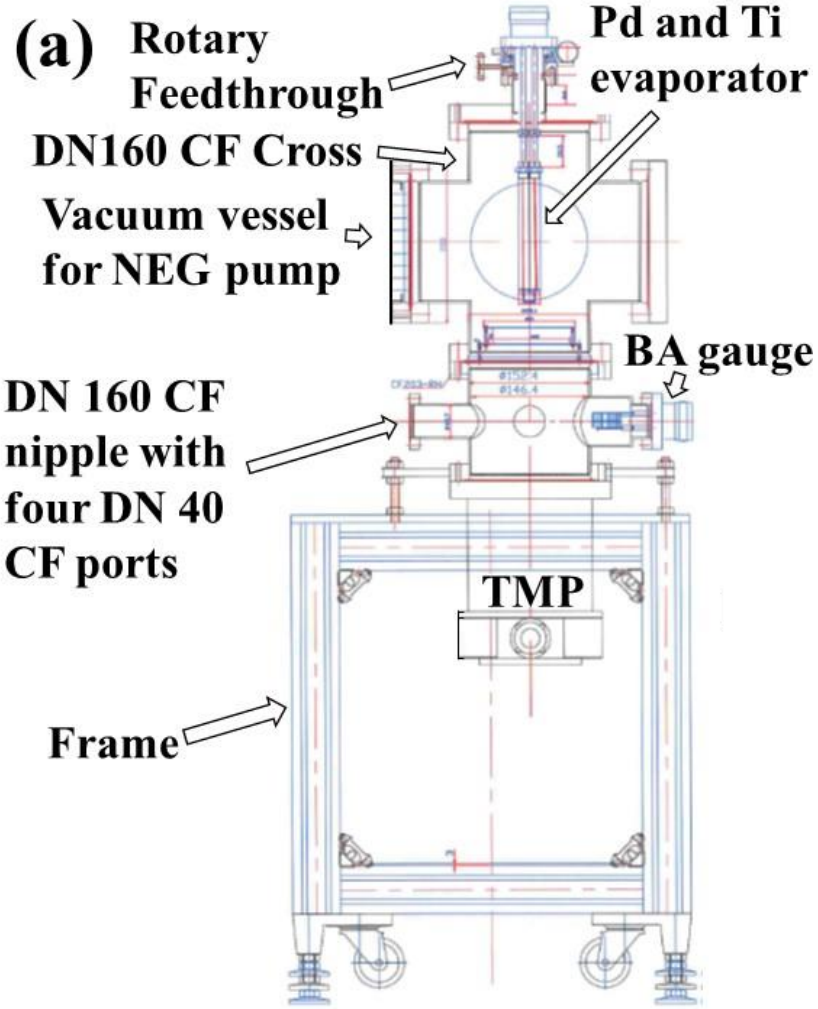
- ✓ **When oxygen-free Pd/Ti thin films are deposited on inner walls of optics chambers and endstations in VSX beamlines, clean UHV can be realized with low cost and low labor.**
- ✓ **However, it is difficult to deposit on the inner walls of existing chambers because optics are installed.**

- ✓ **Therefore, we developed NEG pumps using oxygen-free Pd/Ti.**



BL-13A at PF in KEK

Oxygen-free Pd/Ti thin films were deposited on the vacuum vessel for the zero-length CF fin-type NEG pump by using apparatus shown below.



Pd and Ti evaporator

Procedure

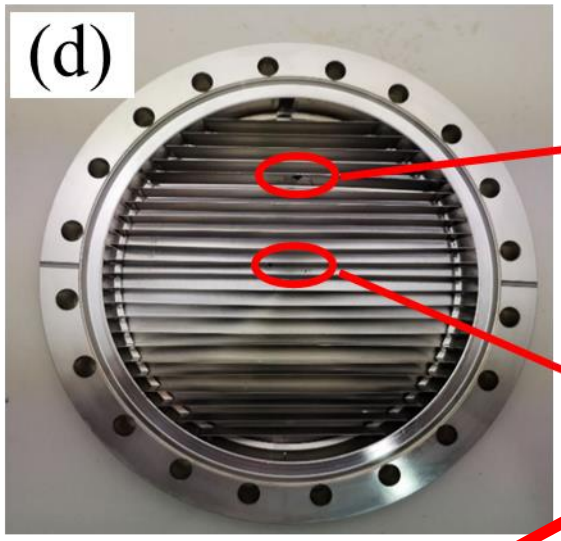
24h baking (Max 150℃)

Degassing
(Ti : 25 A, 1 h,
Pd : 19.5 A, 40 min)

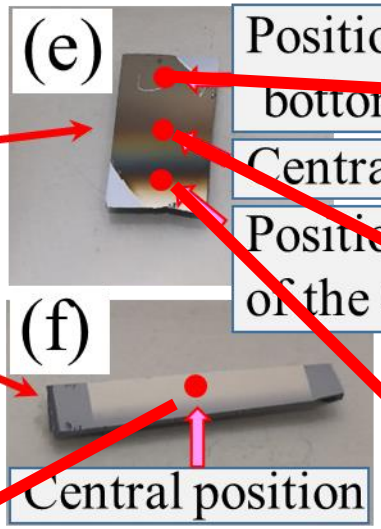
Deposition
(Ti 47.5 A 3h , Pd 33A 5 h)
using deposition rate monitor (Q-pod)

The vacuum vessel for the zero-length CF fin-type NEG pump

Morphologies of oxygen-free Pd/Ti thin films on the fins and the bottom were examined by SEM, STEM, and EDS. The Ti thin film was completely coated with Pd on the bottom, whereas the fins were covered by oxygen-free Pd/Ti nanostructures.



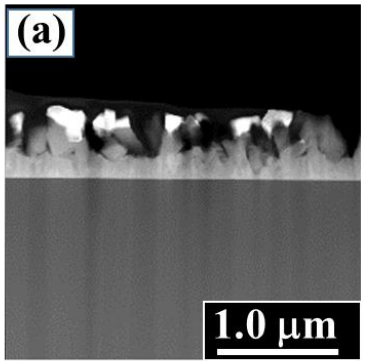
The vacuum vessel

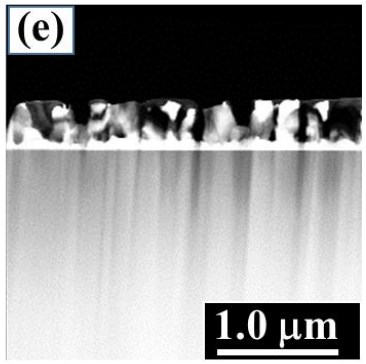


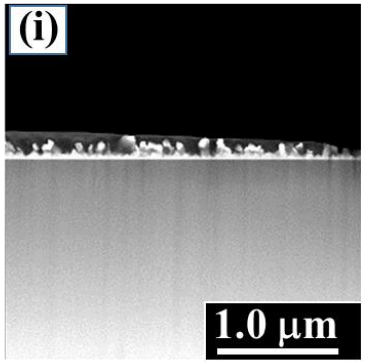
Position distant from the bottom of the CF flange

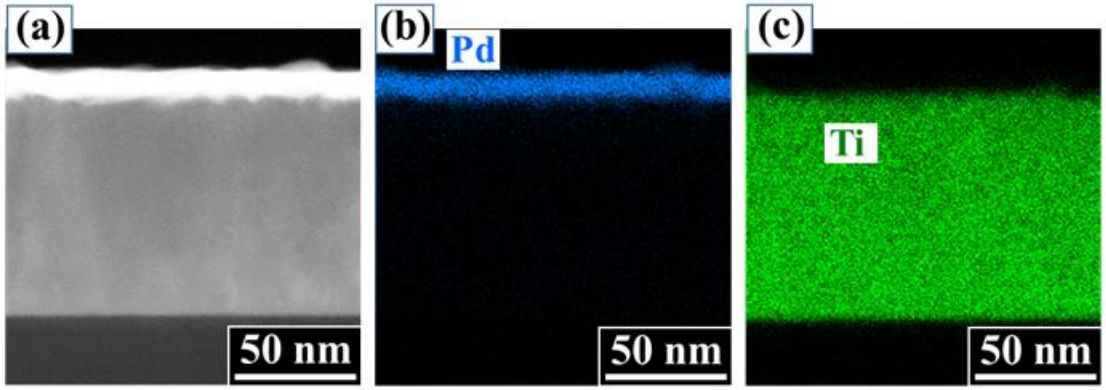
Central position

Position close to the bottom of the CF flange



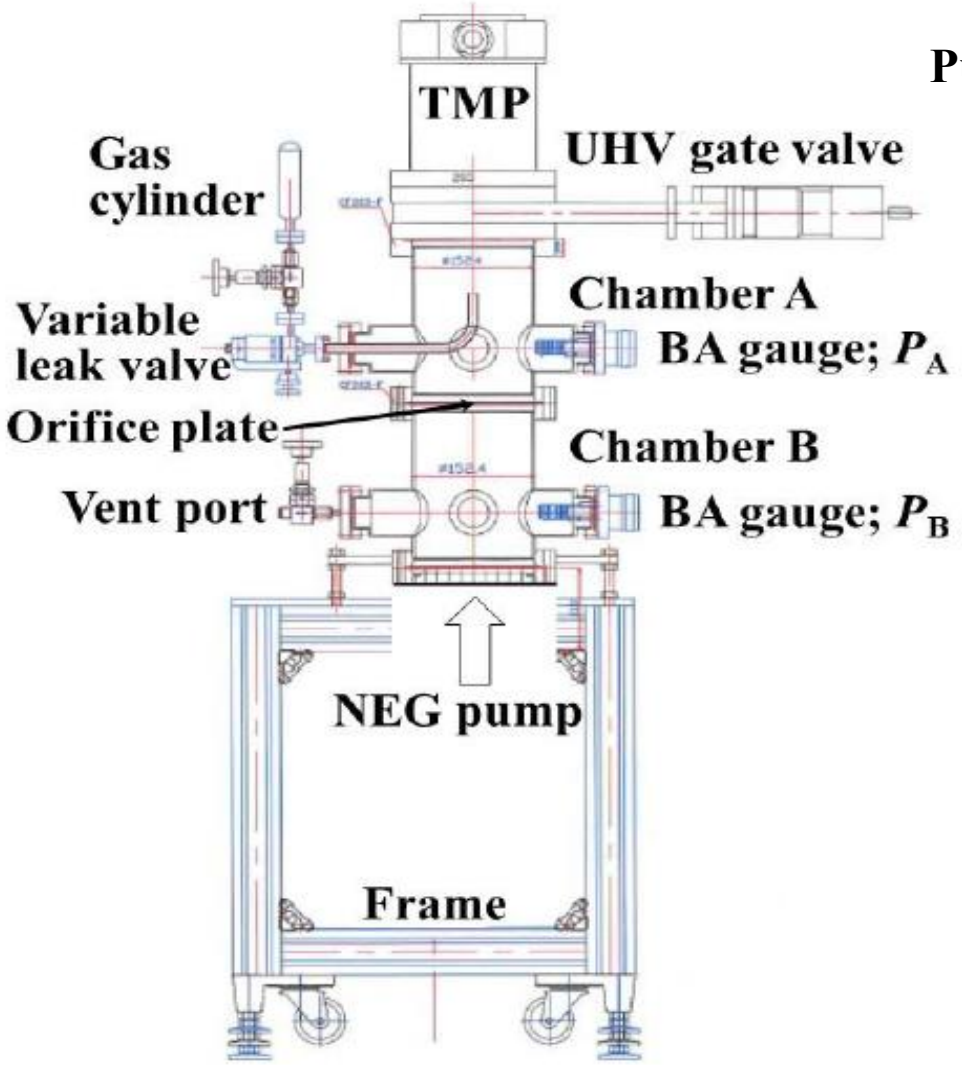






[Y. Sato *et al.*, to be published.]

Pumping speeds of the zero-length CF fin-type NEG pump for H_2 and CO were measured as a function of the pumped quantity using the apparatus shown below.



Pumping Speed measured by Orifice method

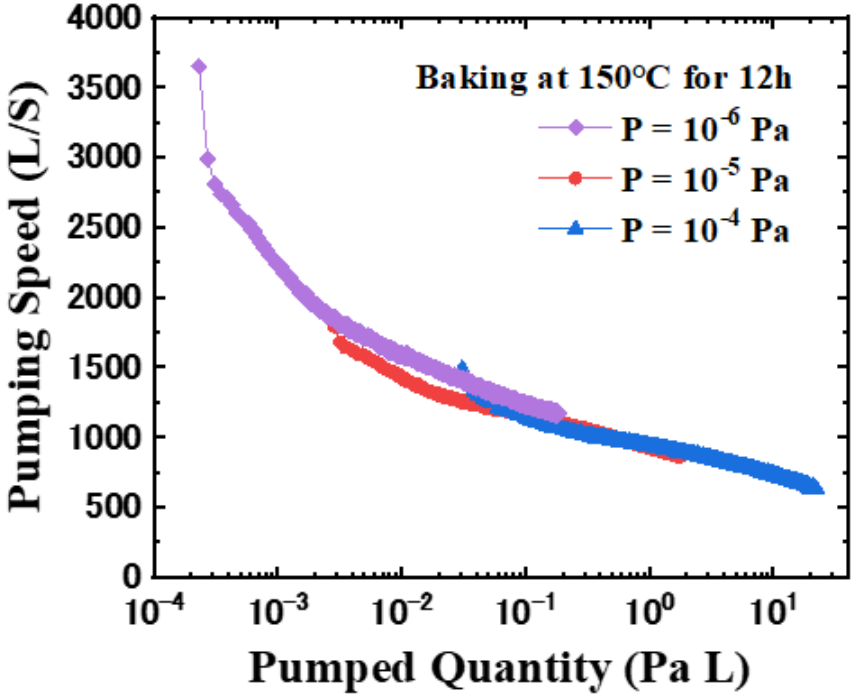
$$S = C\left(\frac{P_a - P_{a0}}{P_b - P_{b0}} - 1\right)$$

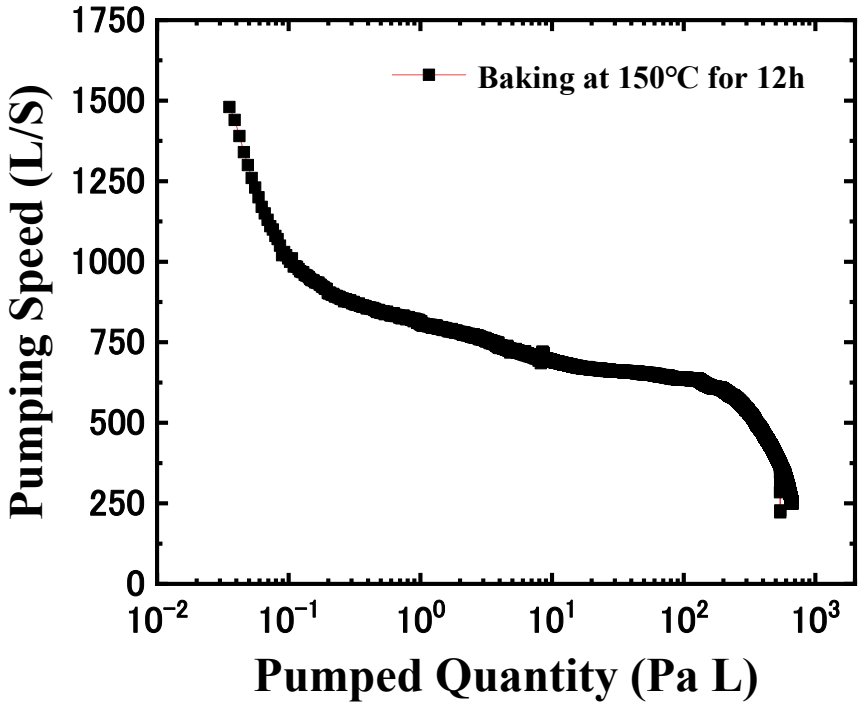
***S* : Pumping Speed of NEG**
***C* : Conductance**
***P* : Pressure in chamber**
P_0 : Base Pressure in chamber

Gas	C (L/s) at 26℃
H_2	33.27
N_2	8.89
O_2	8.31
CO_2	7.09
CO	8.89

[T. Kikuchi *et al.*, AIP Conf. Proc. 2054 (2019) 060046].

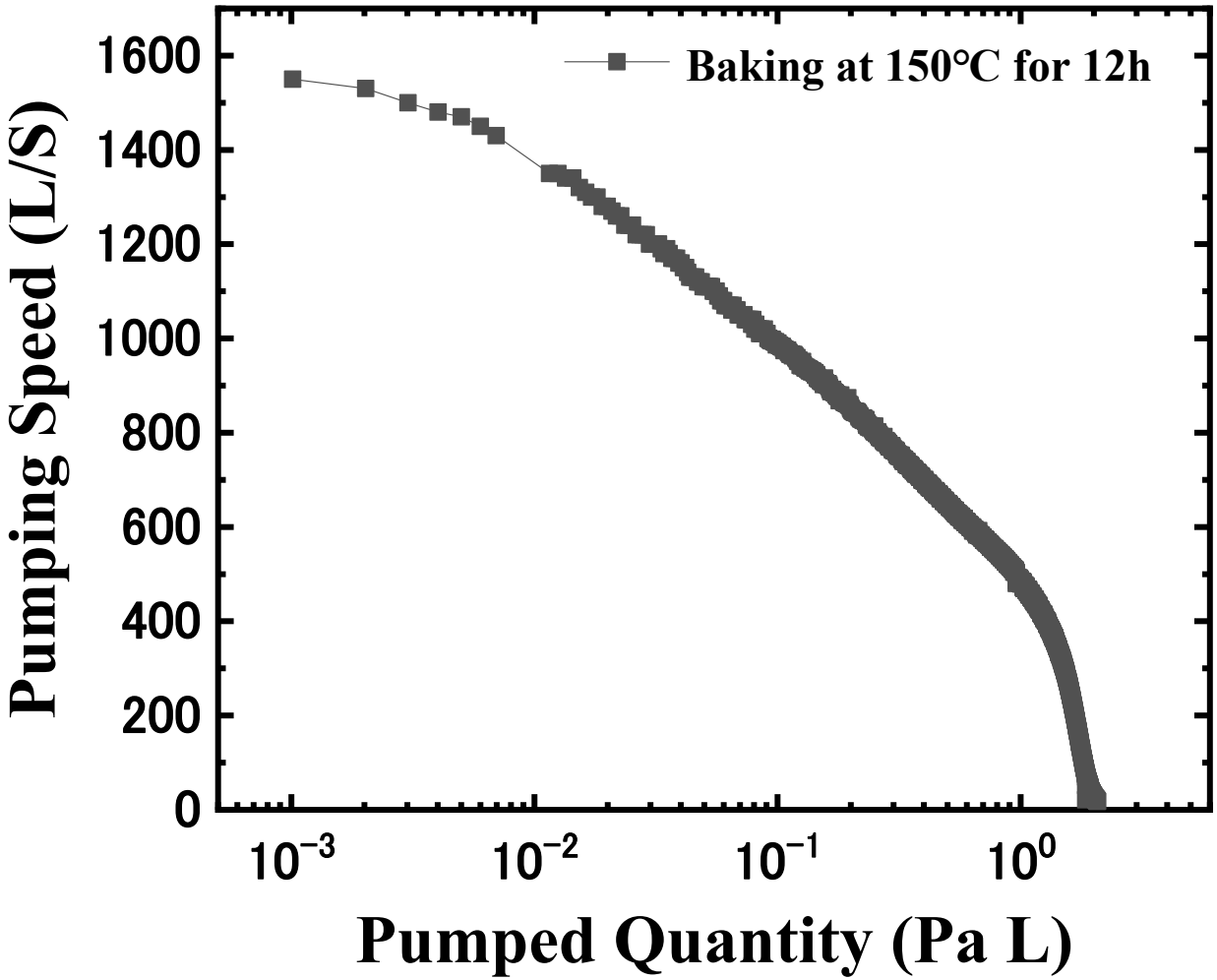
Pumping speeds of the NEG pump using oxygen-free Pd/Ti for H_2 after baking at 150 °C for 12 h





- ✓ The pumping speeds for H_2 were estimated to be about 2800 L s^{-1} at the pumped-quantity of 3 × 10^{-4} Pa L, about 1500 L s^{-1} at the pumped-quantity of 3 × 10^{-3} Pa L, and about 750 L s^{-1} at the pumped-quantity of 10 Pa L.

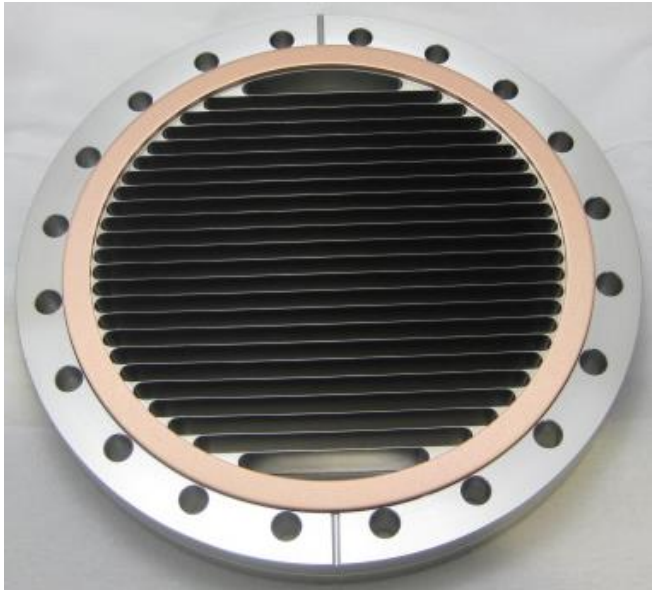
Pumping speeds of the NEG pump using oxygen-free Pd/Ti for CO after baking at 150 °C for 12 h



- ✓ The pumping speed for CO at the pumped-quantity of 1 × 10^{-3} Pa L was about 1550 L s^{-1}.

Commercialization of the zero-length CF fin-type NEG pump using oxgen-free Pd/Ti

- ✓ **Our technology was transferred to Baroque International Inc. and Irie Koken Co., Ltd. The design of the vacuum vessel and the deposition conditions are improved in the joint researches with us. More sophisticated zero-length CF fin-type NEG pump with higher pumping properties have become commercially available now [https://www.baroque-inc.co.jp/custom.html (in Japanese)].**
- ✓ **The commercial NEG pumps are adopted in SR facilities in Japan such as SPring-8, PF, UVSOR, and HiSOR.**
- ✓ **Please send e-mail to mase@post.kek.jp for further information.**



Conclusions

- ✓ **Oxygen-free Pd/Ti deposited vacuum vessel pumps residual H_2 and CO after baking at 150°C for 12 h.**
- ✓ **Pumping speeds of oxygen-free Pd/Ti deposited vacuum vessels for H_2 and CO do not decrease even after repeated activation and exposure to air because Pd over layer prevents Ti film from oxidation.**
- ✓ **Outgassing of H_2, H_2O, CH_4, and CO are suppressed in the case of oxygen-free Pd/Ti coated vacuum vessels even after baking at 100°C for 1 h.**
- ✓ **Economy NEG pumps using oxygen-free Pd/Ti are commercially available now.**
- ✓ **Please send e-mail to mase@post.kek.jp for further information.**

Acknowledgements

This work was partly supported by the Matching Plan-ner Program from the Japan Science and Technology Agency (JST) (VP29117940903), Grants-in-Aid for Scien-tific Research (JSPS KAKENHI Grant Nos. JP17K05067 and JP19K05280), TIA-Kakehashi grants (TK18-014 and TK19-035), joint research with Baroque International Inc. (18C208), joint research with Irie Koken Co., Ltd. (18C220), and the 2019 Takahashi Industrial Economic Research Foundation research grant (08-003-172). We would like to thank Mr. Tomohiro Okada (Tokyo Gaku-gei Univ.) and Mr. Hiromu Nishiguchi (Baroque Interna-tional Inc.) for their invaluable advice and support. Partial pressure measurements were carried out as a collaborative study with Osaka Vacuum, Ltd. We are grateful to T. Iga, T. Koyama, S. Sugimoto, and M. Iguchi (Osaka Vacuum, Ltd.) for their invaluable support.

Thank you for your attention!