Zero-Length Conflat Flange Nonevaporable Getter (NEG) Pump Manufactured by Oxygen-Free Pd/Ti Deposition

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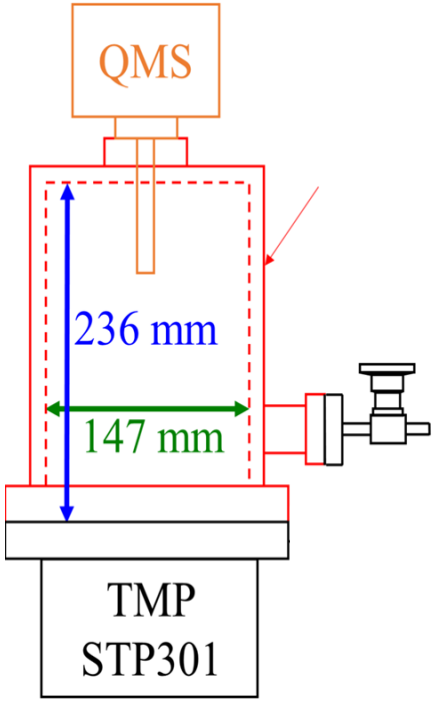
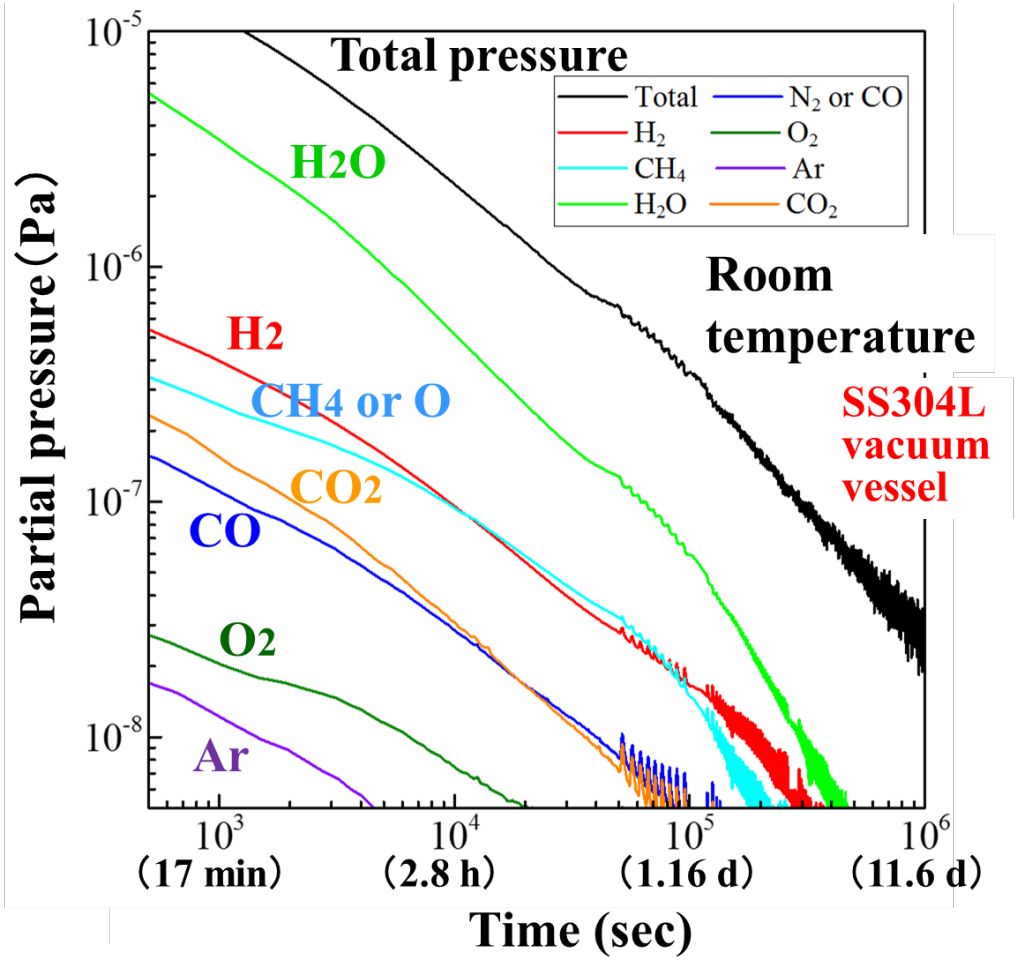
1. Introduction -Why does pumping takes long time at RT?

Prisma 80;
Pfeiffer
Vacuum





- ✓ **To achieve hydrocarbon-free ultra high vacuum (UHV) for SX beamlines and end stations baking at 80 ~ 150 ℃ is required.**
- ✓ **My presentation will focus on the vacuum technology to easily realize a hydrocarbon-free UHV of 1×10^{-8} Pa.**

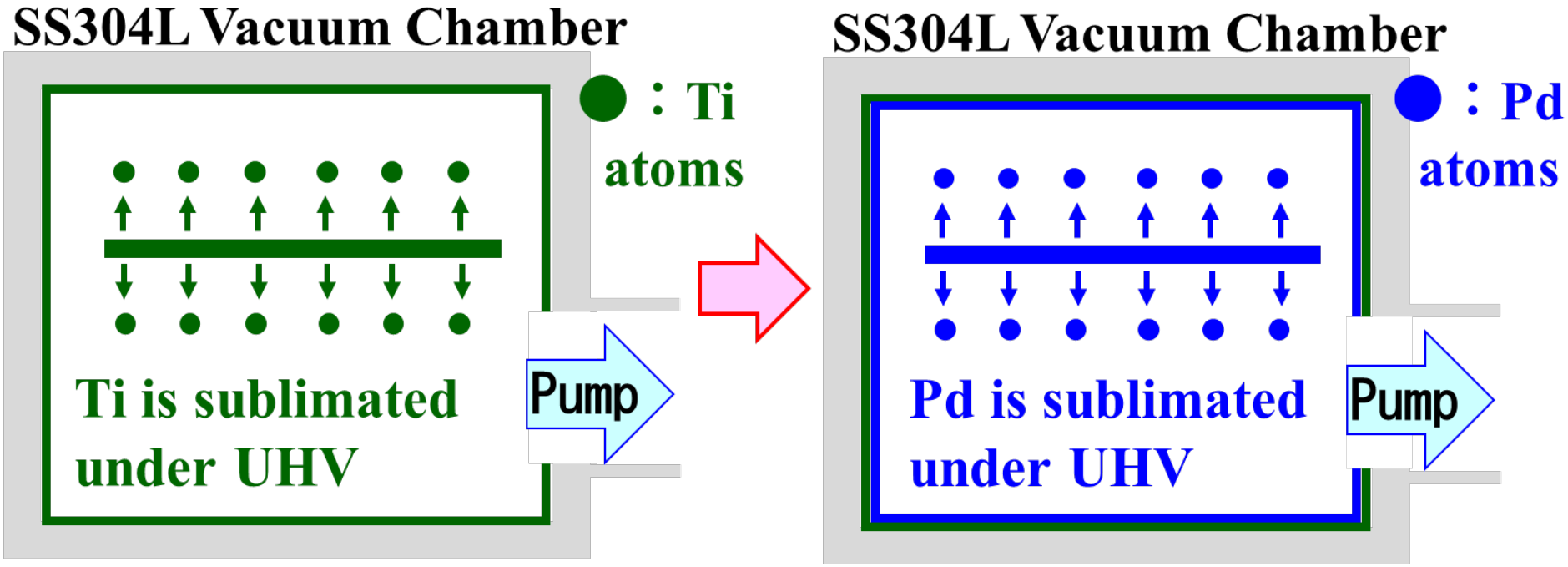
UHV technology required for SX beamlines and end stations

- ✓ In the initial pumping and baking to achieve hydrocarbon-free UHV for SX beamlines and end stations, dry pumps (DPs) and turbomolecular pumps (TMPs) are required.
- ✓ To maintain hydrocarbon-free 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 and noise.
- ✓ Hydrocarbons in the SX beamline should be removed to suppress carbon contamination on the gratings and mirrors.

- ✓ 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 75 ~ 150 ℃ for 6 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. Thus hydrocarbons can be removed from beamline.

2. Oxygen-free Pd/Ti deposition

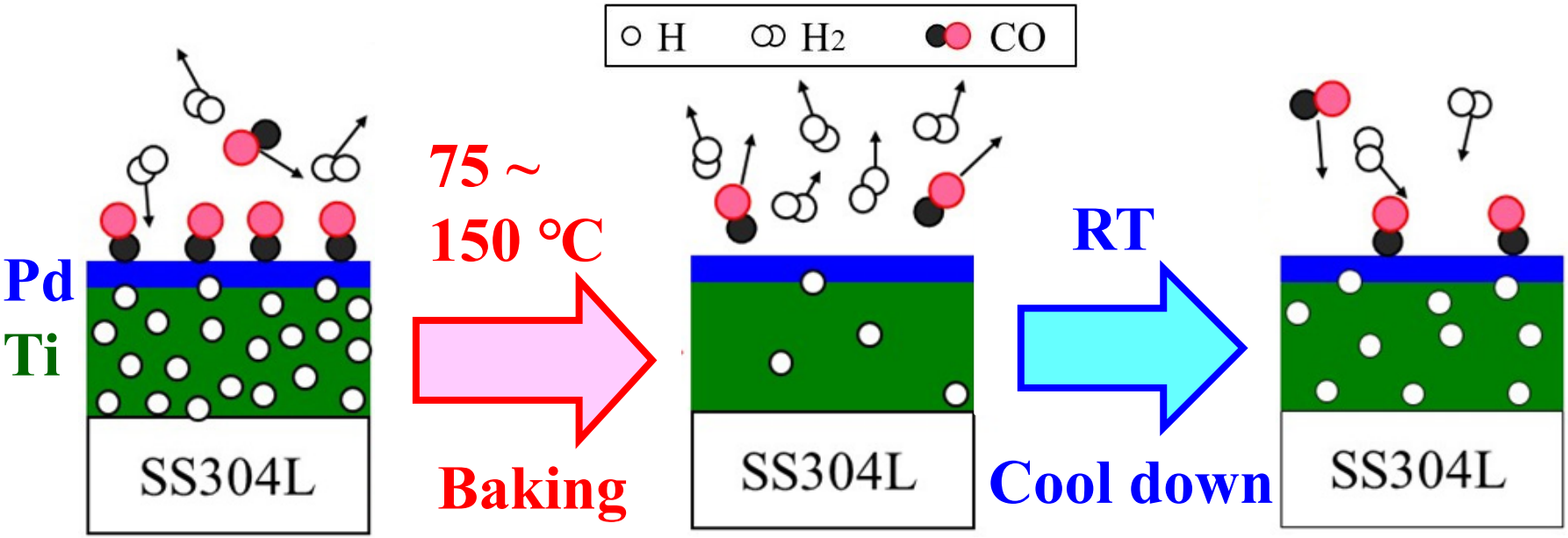
Oxygen-free Pd/Ti thin films were deposited by sequential sublimation of Ti and Pd under UHV in range 10^{-7} to 10^{-8} Pa. This Pd/Ti was named oxygen-free Pd/Ti, because its oxygen content was estimated to be less than 0.05% .



K. Mase and T. Kikuchi, WO/2018/097325 (May 31, 2018).
[T. Miyazawa *et al.*, J. Vac. Sci. Technol. A 36, 051601 (2018).]

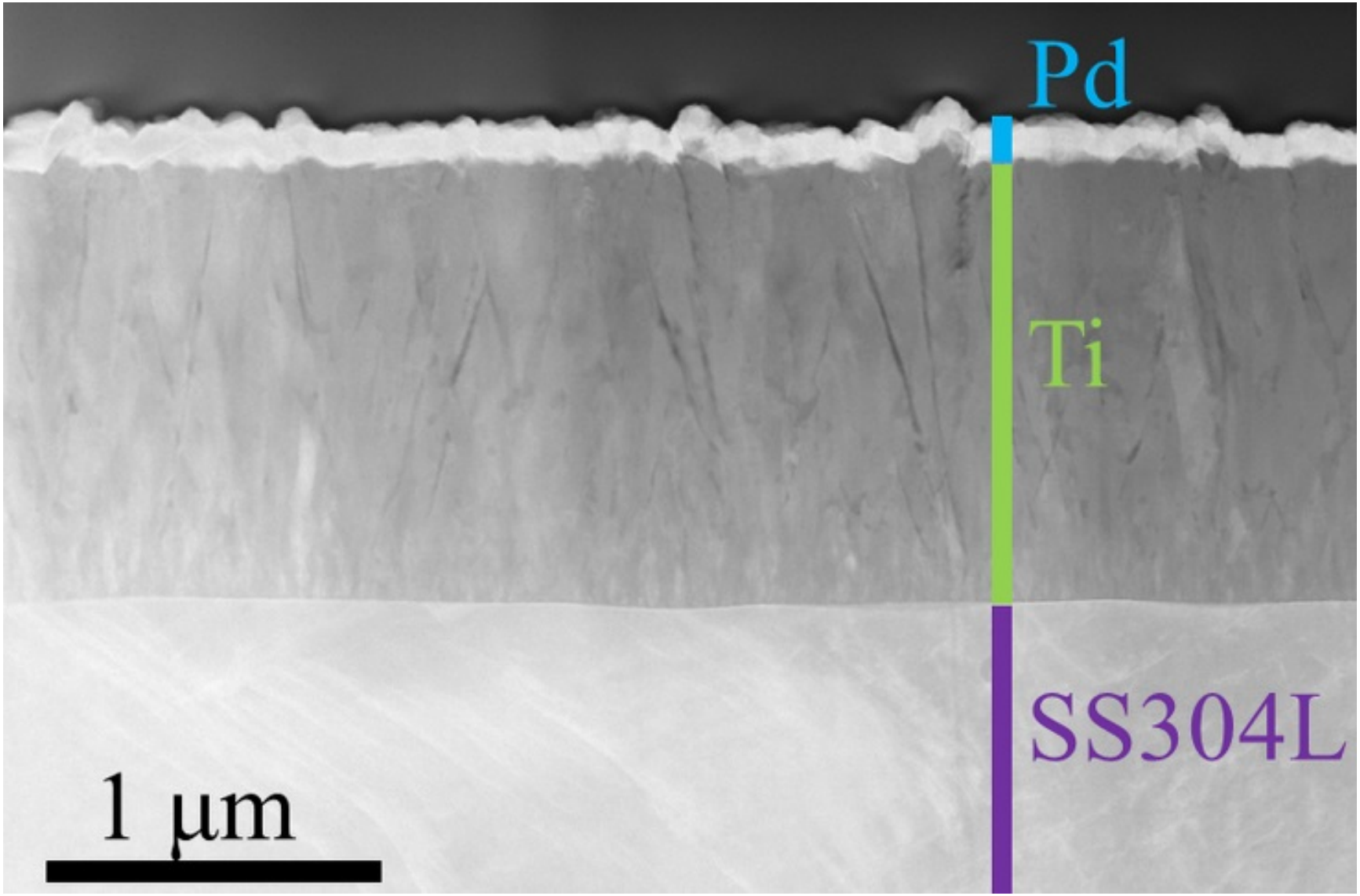
Activation & pumping mechanisms of oxygen-free Pd/Ti

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



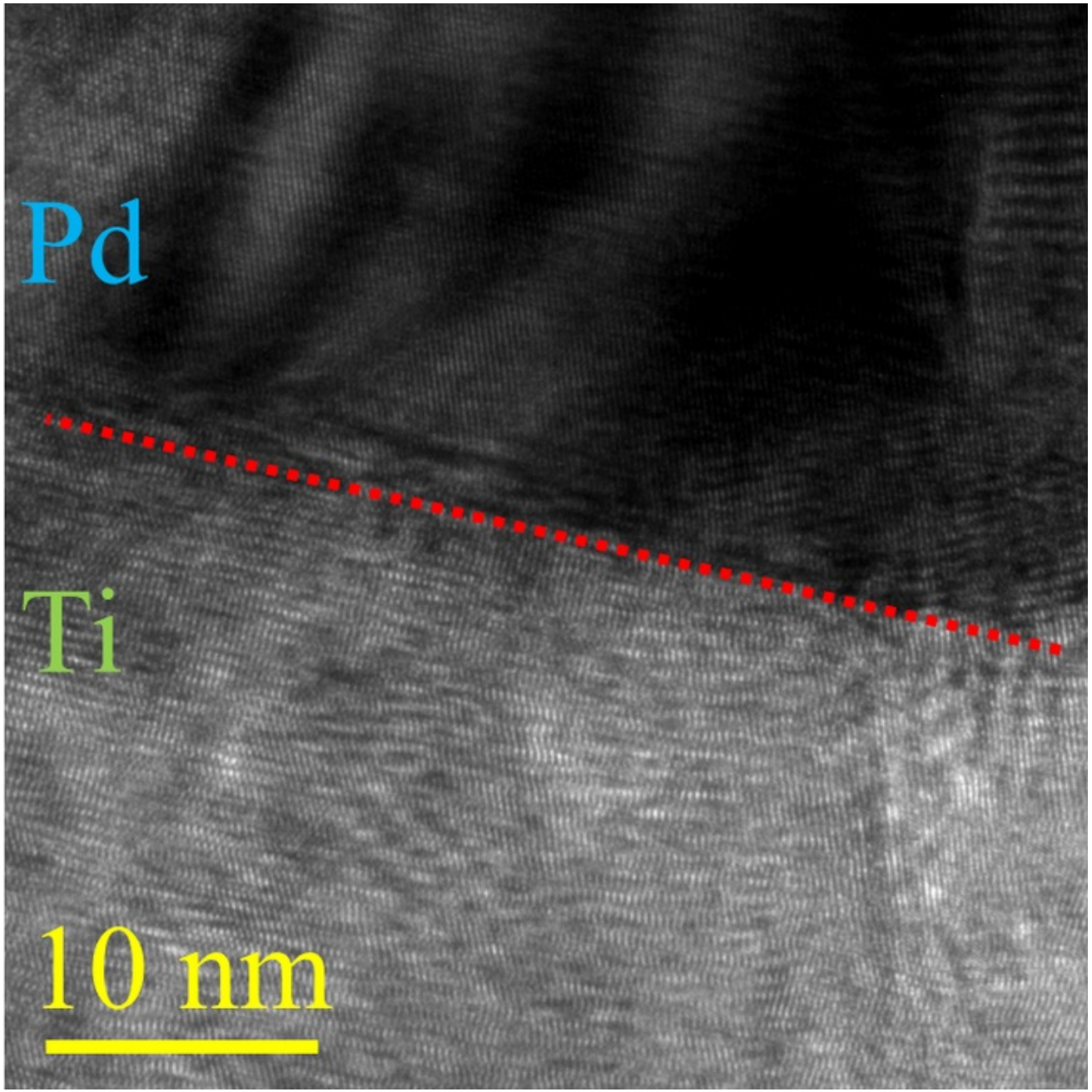
[T. Kikuchi *et al.*, AIP Conf. Proc. 2054 (2019) 060046].

STEM dark-field image of cross section of oxygen-free Pd/Ti



This STEM image was obtained at NIMS at an accelerating voltage of 200 kV. [T. Miyazawa *et al.*, JVSTA 36, 051601 (2018).]

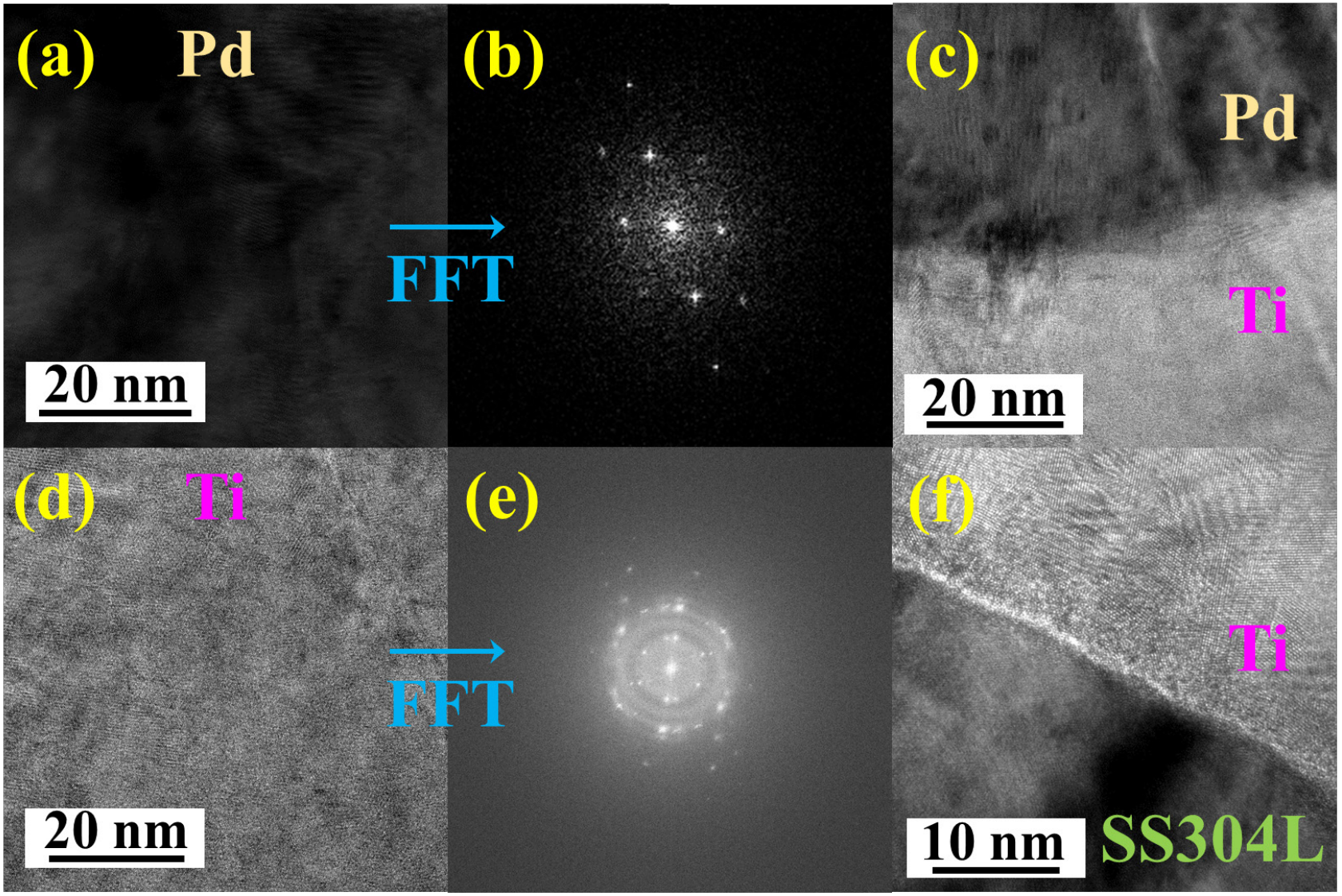
TEM bright-field image of interface between Ti & Pd



- ✓ No TiO_2 layer at the Pd/Ti interface.

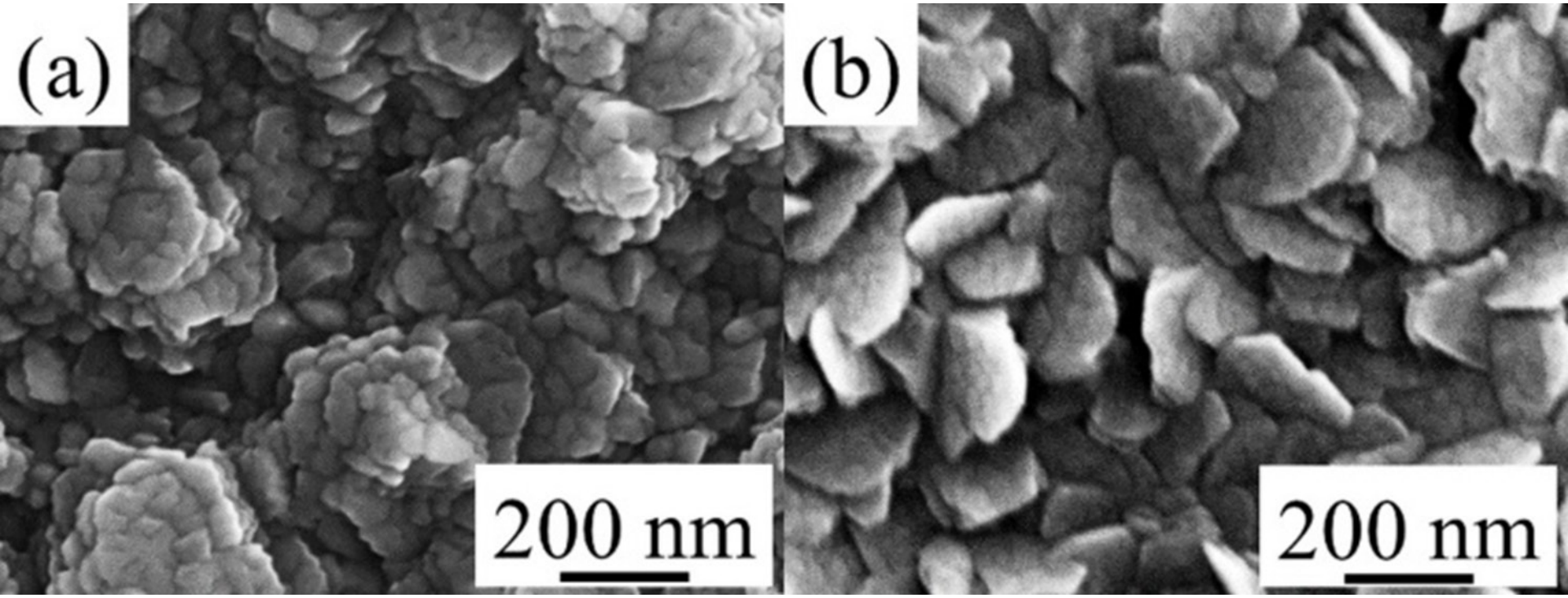
This TEM image was obtained at NIMS using a JEM-2100F (JEOL Ltd.) operating at an accelerating voltage of 200 kV. [T. Miyazawa et al., JVSTA 36, 051601 (2018).]

TEM bright-field image of interface between Ti & Pd



✓ Ti and Pd thin films are polycrystalline. This images were obtained at NIMS [T. Miyazawa *et al.*, MEDSI2018 proceedings, TUPH25 (2018).]

SEM images of surface of oxygen-free Pd/Ti



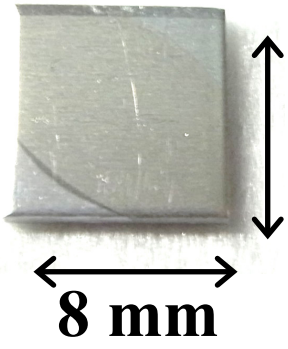
Before heating at 150 ℃ for 12 h **After heating at 150 ℃ for 12 h**

This SEM image was obtained at NIMS at an accelerating voltage of 15 kV. [T. Miyazawa, et al., JVSTA 36, 051601 (2018).]

- ✓ **H_2 is known to be dissociatively chemisorbed at the stepped sites of Pd surfaces such as Pd (110) surface while CO is known to be molecularly chemisorbed on Pd (111) surface.**

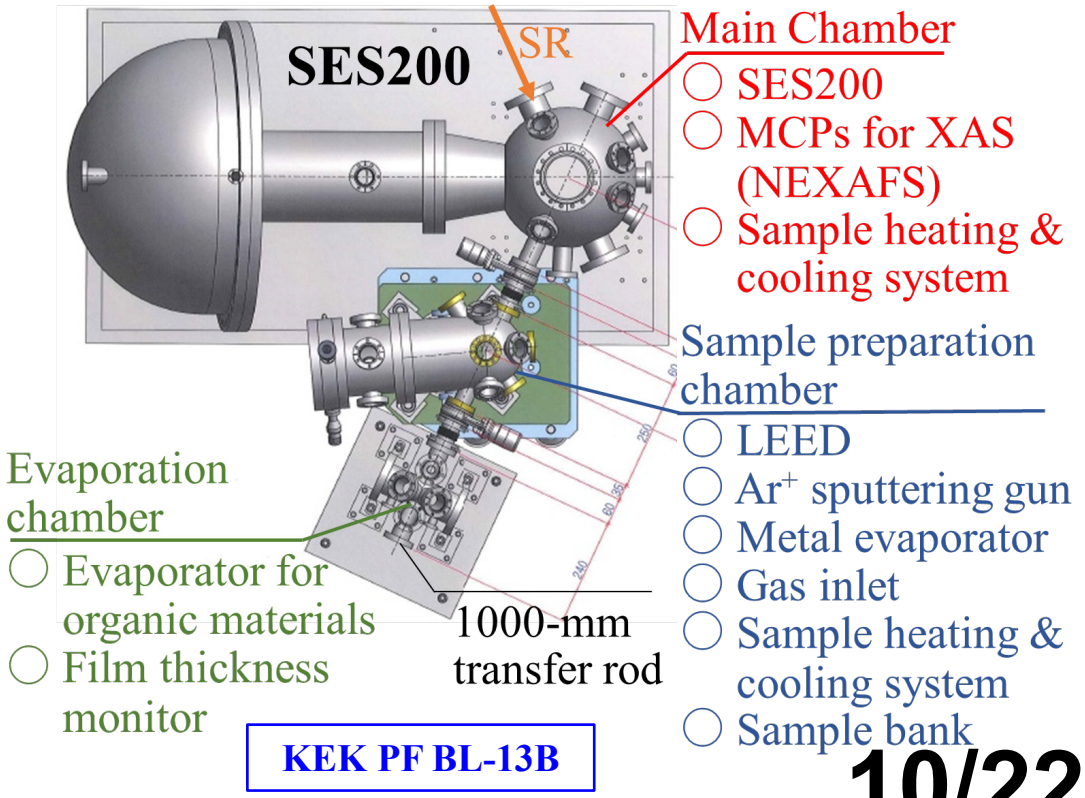
SR-XPS measurements of oxygen-free Pd/Ti at PF

Oxygen-free
Pd(50 nm)/Ti(1 μm)
deposited on SS304L

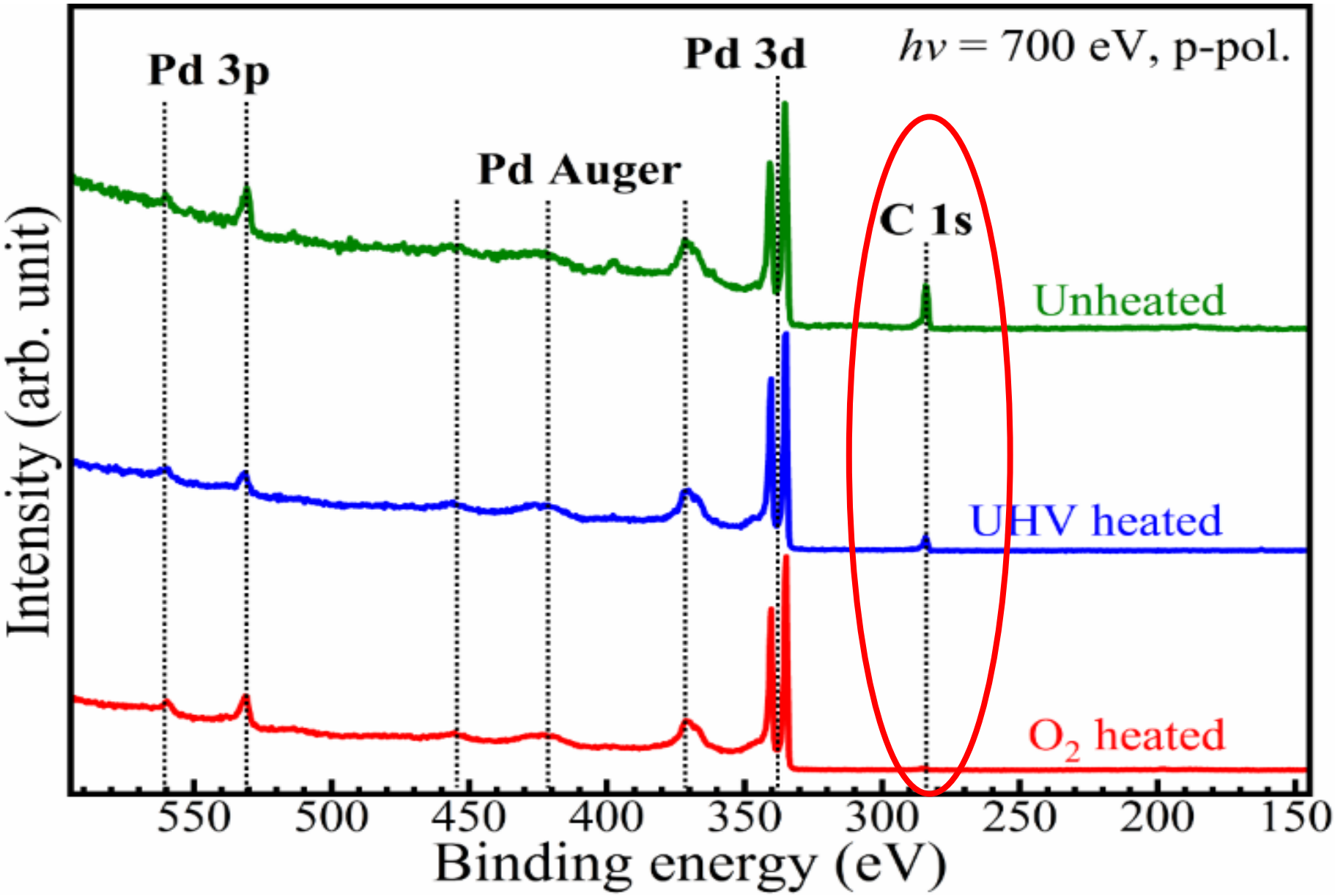


1. **Unheated**
2. **Heated in UHV at 150℃ (UHV heated)**
3. **Heated under an O_2 pressure of 1.3×10^{-4} Pa (O_2 heated)**

Surface of Oxygen-free Pd(50nm)/Ti(1μm) was analyzed by XPS using SR with *hv* = 700 eV at BL-13B in Photon Factory, KEK



SR-XPS spectra of oxygen-free Pd/Ti



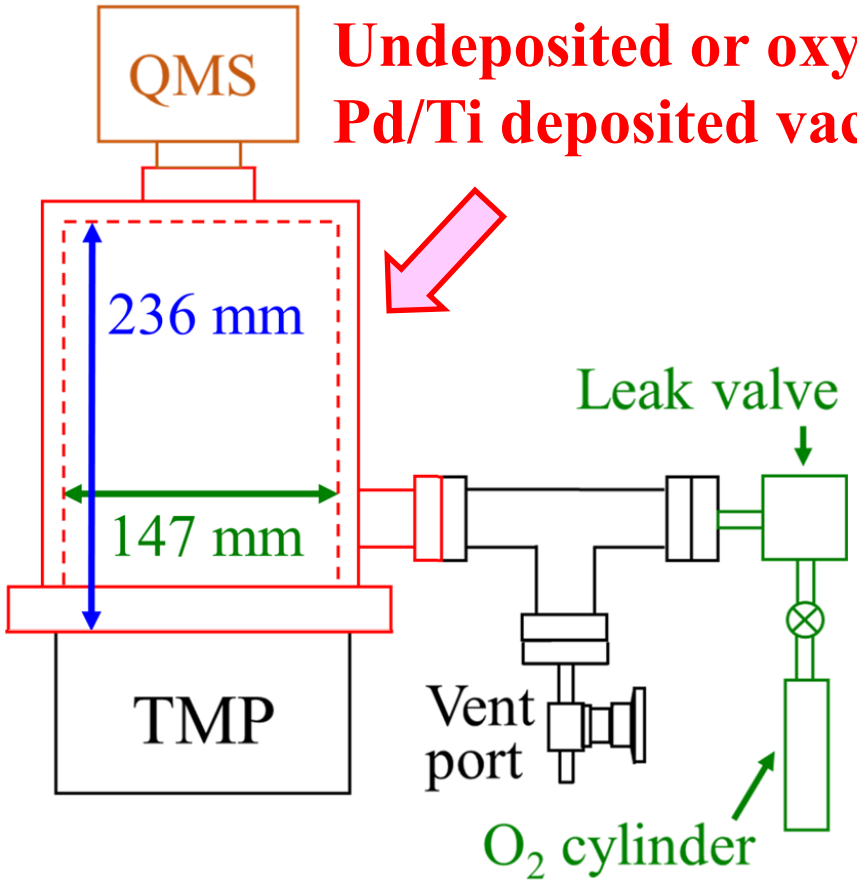
[T. Miyazawa *et al.*, J. Vac. Sci. Technol. A 37, 02160 (2019)]

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

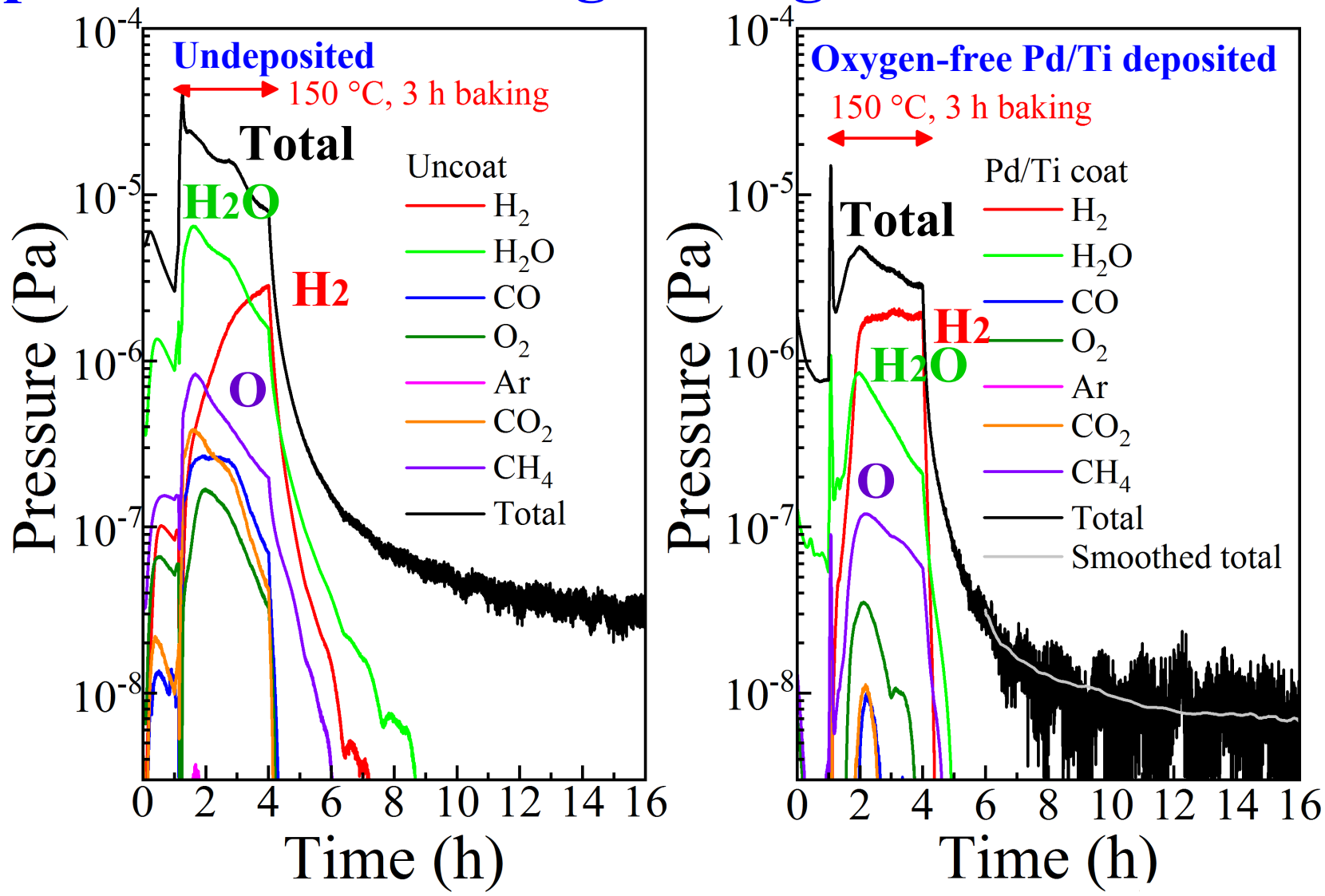
Total pressure was also measured with the QMS.



Pumpd by RP + FT + TMP

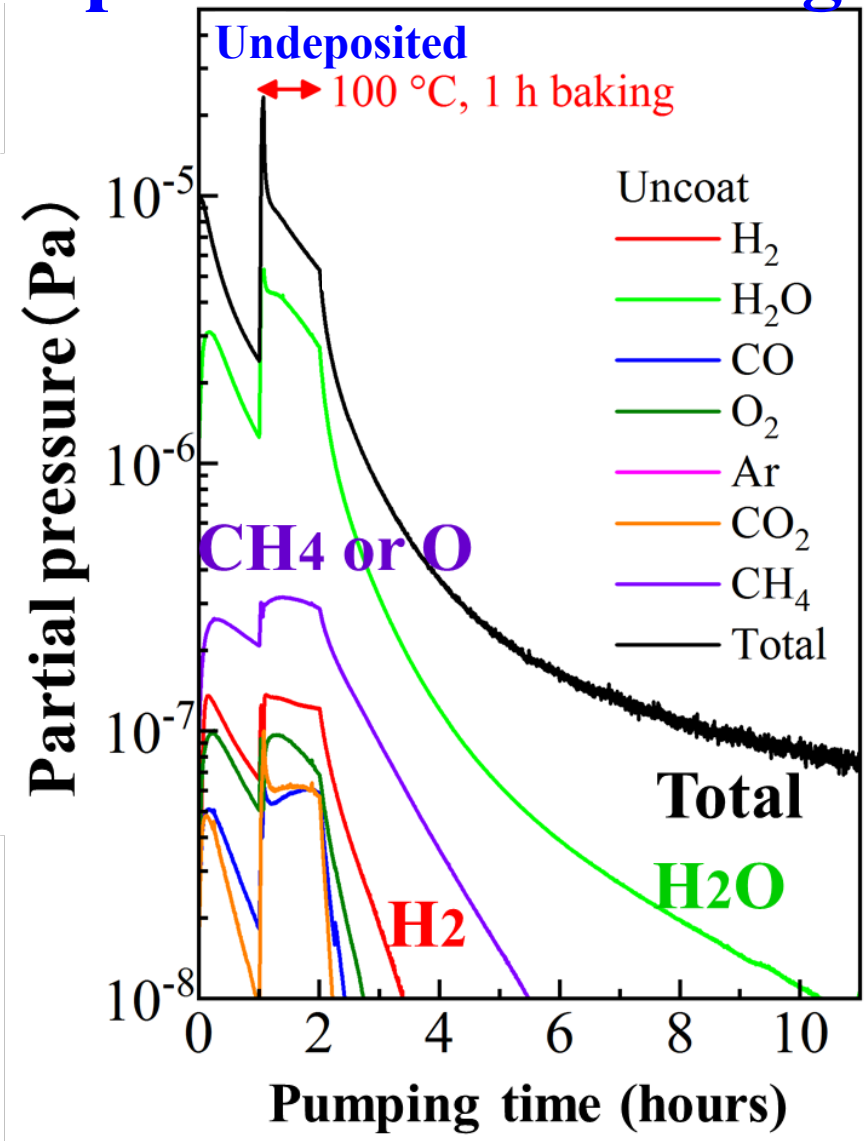
[T. Miyazawa *et al.*, to be published].

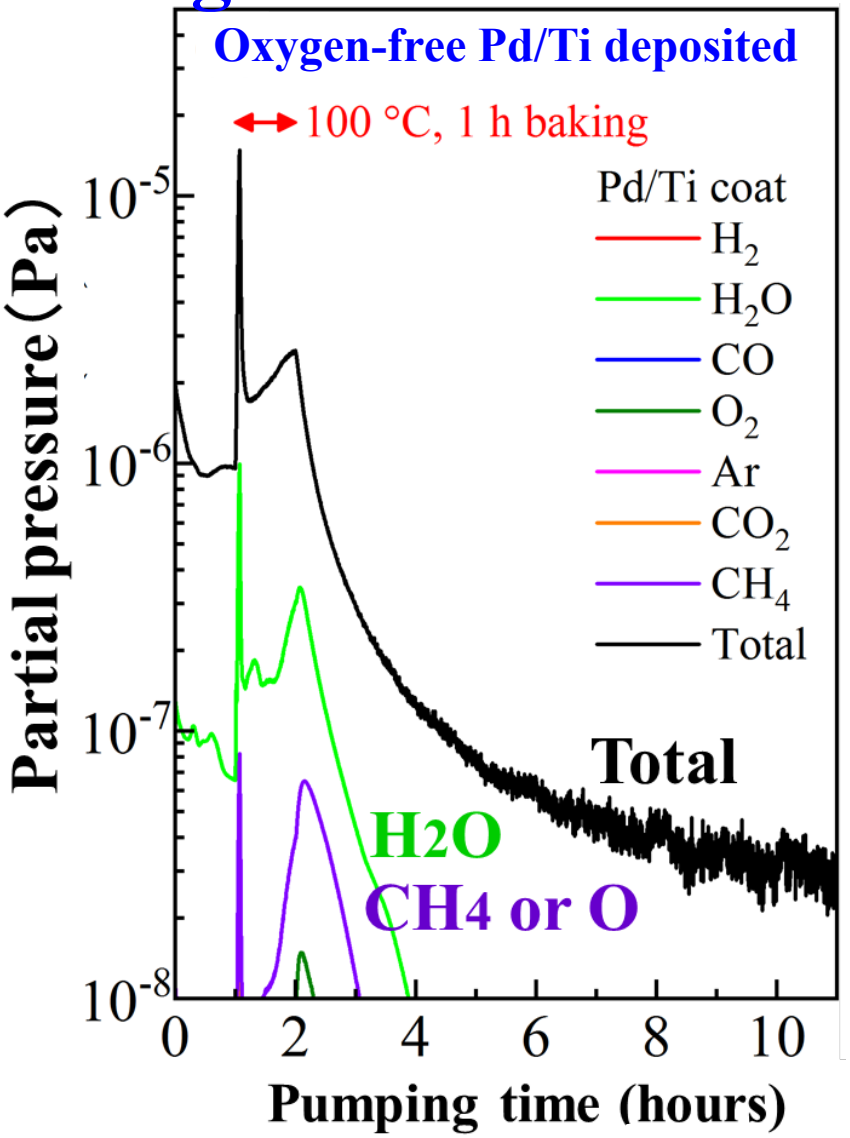
Pressure curves of uncoated and oxygen-free Pd/Ti deposited vessel during baking at 150 °C for 3 h



[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

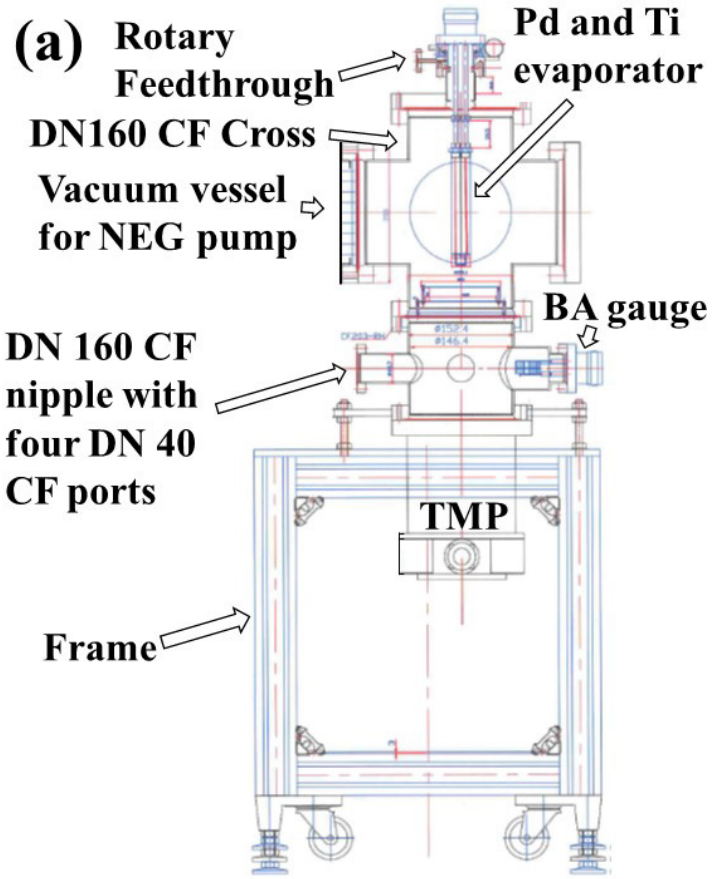


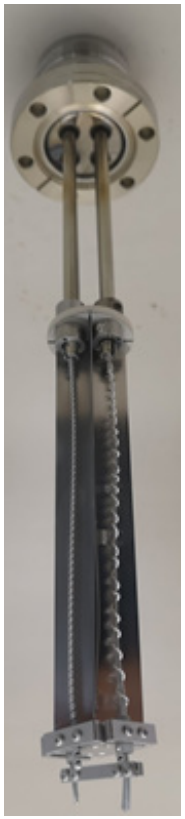


[T. Miyazawa *et al.*, to be published].

3. NEG Pump using oxygen-free Pd/Ti deposition

We have developed zero-length ICF NEG pump by using oxygen-free Pd/Ti deposition.





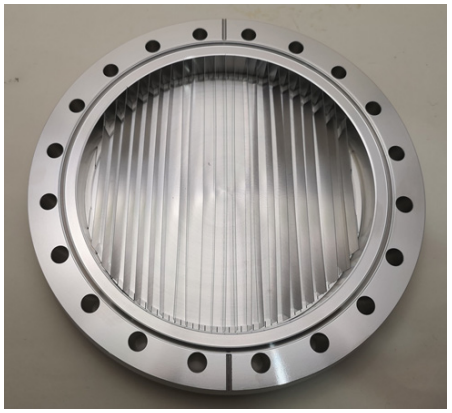
Pd and Ti evaporator

Procedure

24h baking (Max 150°C)

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

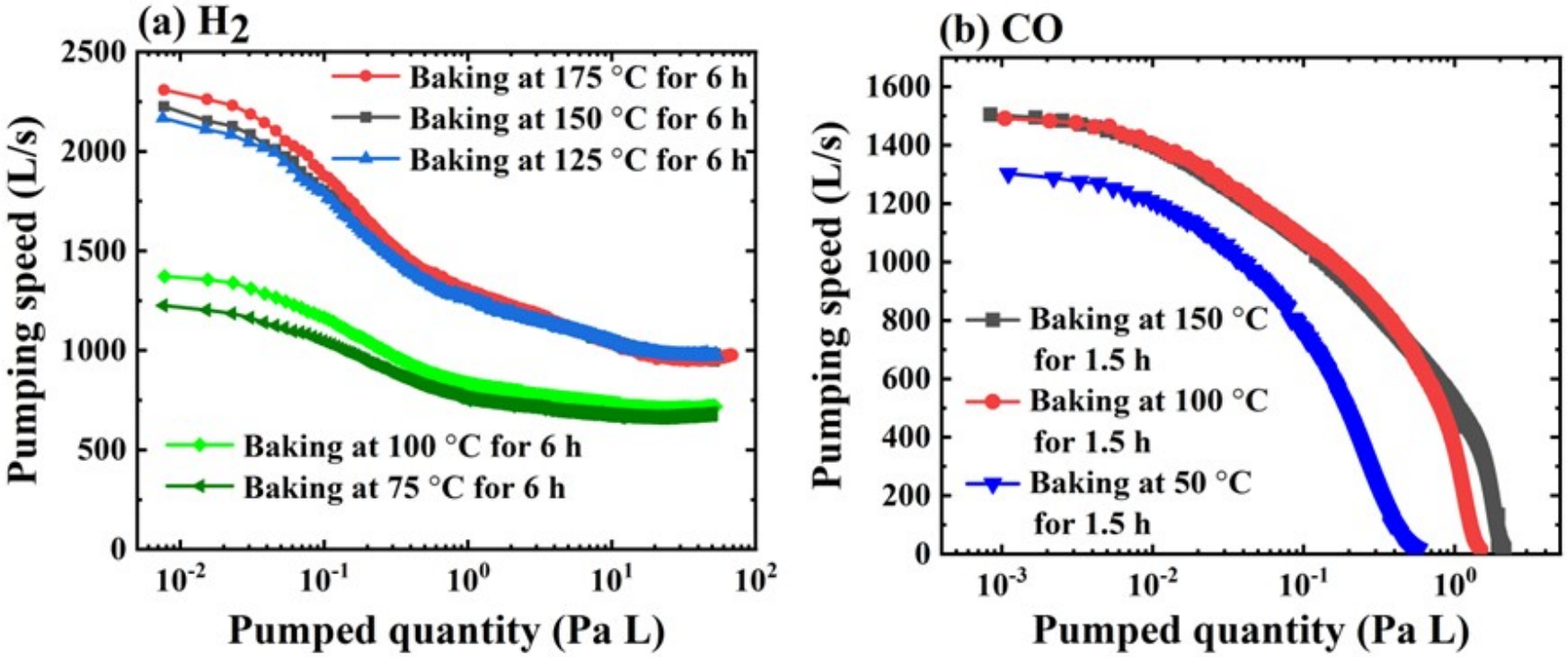
Deposition
(Ti 47.5 A 3h , Pd 33A 5 h)
using deposition rate monitor (Q-pod)



The vacuum vessel for the zero-length ICF 203 NEG pump

[Y. Sato *et al.*, Vacuum 212, 112004 (2023).]

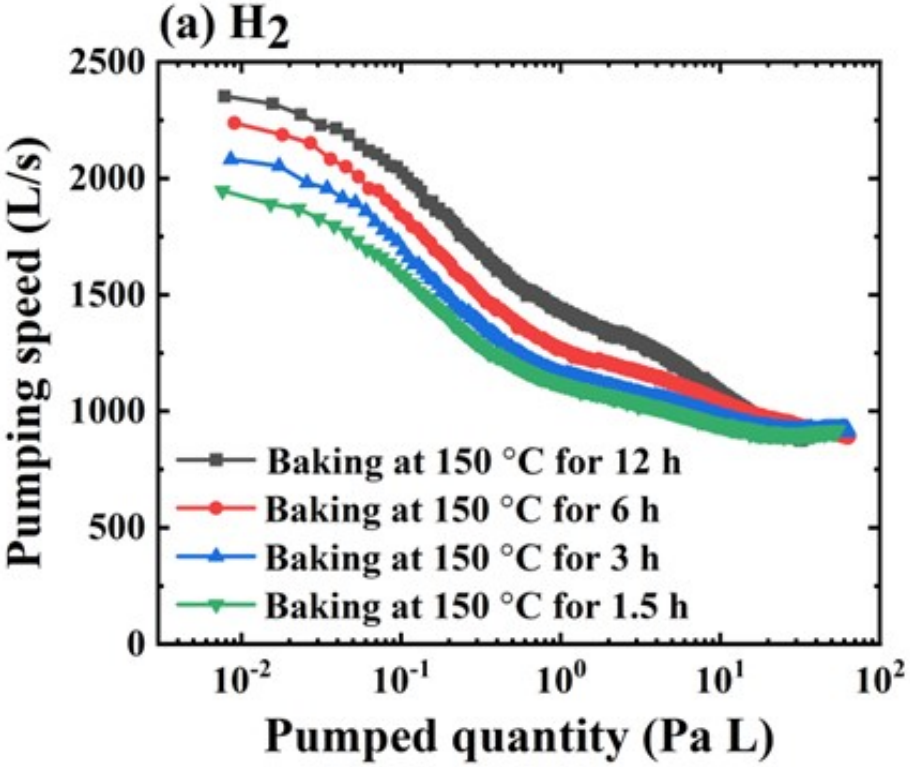
Pumping speeds of the NEG pump using oxygen-free Pd/Ti for H_2 and CO − Temperature dependence −

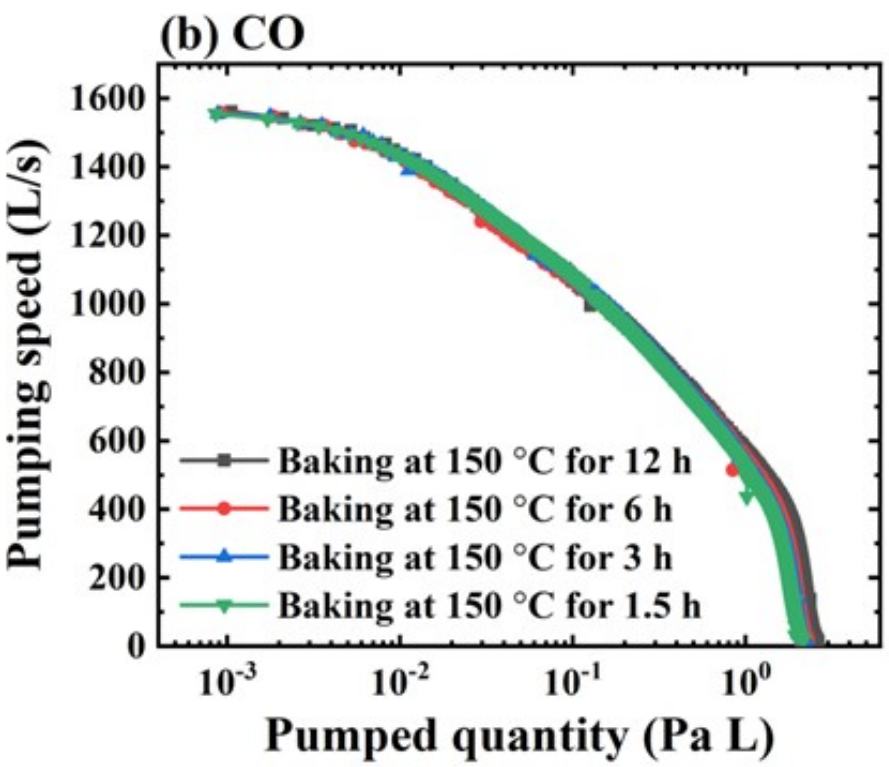


- ✓ **The NEG pump can be fully activated by baking at 150 ℃ for 6 h and exhibits initial pumping speeds of 2340 L/s for H_2 and 1440 L/s for CO.**
- ✓ **The NEG pump can be partially activated by baking at 75 ℃.**

[Y. Sato *et al.*, Vacuum 212, 112004 (2023).]

Pumping speeds of the NEG pump using oxygen-free Pd/Ti for H_2 and CO − Baking time dependence −



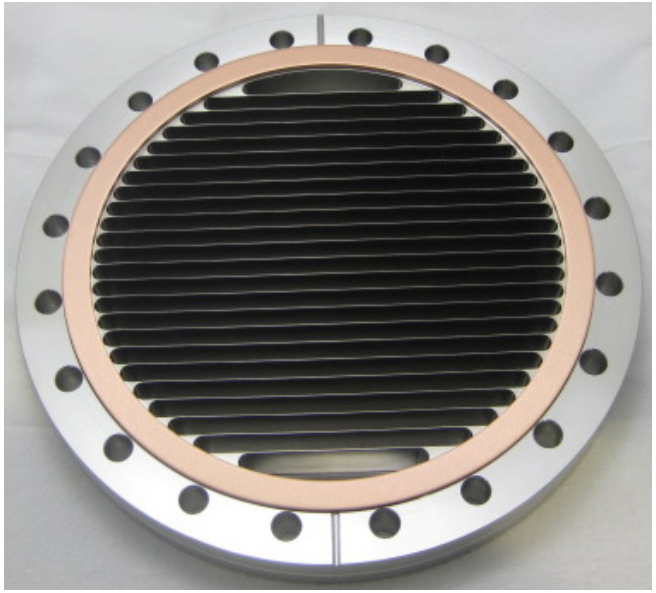


- ✓ **Baking at 150 ℃ for 1.5 hours recovers 80% of pumping speeds of the NEG pump using oxygen-free Pd/Ti deposition.**

[Y. Sato *et al.*, Vacuum 212, 112004 (2023).]

Commercialization of the zero-length ICF NEG pump using oxygen-free Pd/Ti deposition

- ✓ **Our technology was transferred to two private companies. The design of the NEG pump and the deposition conditions are improved in the joint researches with us. More sophisticated zero-length ICF NEG pump with higher pumping properties have become commercially available from 2018 in Japan. More than 90 NEG pumps have been delivered to date.**
- ✓ **The commercial NEG pumps are widely used in SR facilities such as SPring-8, UVSOR, PF, etc., universities, and private companies in Japan.**
- ✓ **Efforts to expand overseas sales channels have also begun.**
- ✓ **We have also begun joint research for application to semiconductor-related industries.**



Comparison of present NEG pump with conventional one

	Capaci Torr® D 2000	Present NEG pump
Composition of NEG	ZrVFe	Oxygen-free Pd/Ti
Coating method	Sintered	Deposition under UHV
Principle of activation	Diffusion of surface O, Desorption of H_2	Desorption of H_2 and CO
Pumping speeds for H_2	2000 L/s	2340 L/s (initial, 150 ℃)
Pumping speeds for CO	1000 L/s	1440 L/s (initial, 150 ℃)
Gasses that can be pumped	H_2, H_2O, N_2, CO, CO_2	H_2, CO
Pumping speeds after frequent air vent	Pumping speeds do not decrease after 450 °C-activation-venting cycles	Pumping speeds do not decrease after 150-°C-activation-venting cycles
Activation temp. & time	450°C for 10 min	75 ~ 150 °C for 1.5 ~ 6 h
Costs for manufacturing	-	Relatively low
Dedicated power supply	Required	Not required
Current feedthroughs	Required	Not required
Space required	Relatively large	Zero-length
Getter mass	225 g	< 0.5 g
Adopt to SR facilities	Adopted around the world	Not yet

✓ Please decide which NEG pump to choose for your vacuum system in consideration of the merits and demerits of them.

4. Prospects for future

Oxygen-free Pd/Ti deposition is a sustainable vacuum technology to maintain hudrocarbon-free UHV without electric power except for baking. Our final goal is to contribute to the SDGs by developing new vacuum technologies that can be applied not only to SR facilities but also to industry.

https://ja.wikipedia.org/wiki/持続可能な開発目標/

5. Summary

- ✓ **A new NEG material, oxygen-free Pd/Ti, has been developed and patented.**
- ✓ **Oxygen-free Pd/Ti thin film can pump H_2 and CO at room temperature after baking at 75 ~ 150 ℃ for 6 hours. Hydrocarbons can be removed at the same time.**
- ✓ **Zero-length ICF NEG pump using oxygen-free Pd/Ti deposition has been developed.**
- ✓ **Improved NEG pumps using oxygen-free Pd/Ti deposition are commercially available now.**
- ✓ **Our final goal is to contribute to the Sustainable Development Goals (SDGs) by developing new vacuum technologies that can be applied not only to SR facilities but also to industry.**

Acknowledgements

I would like to thank collaborators: Mr. Takashi Kikuchi, Prof. Kenichi Ozawa (KEK-IMSS-PF), Dr. Tetsuya Miyazawa (SOKENDAI), Mr. Masashi Kurihara, Mr. Yuta Sato, Prof. Shinya Ohno (Yokohama Nat. Univ.), Ms. Ayako Hashimoto, Ms. Misao Yamanaka (NIMS), Mr. Hiromu Nishiguchi (Baroque International Inc.) Yoshihiro Kato, Tadayoshi Ogawa, Manabu Yabe (Irie Koken Co., Ltd.) Toshiharu Iga (Osaka Vacuum, Ltd.) for his invaluable advice and support.

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Thank you for your attention!

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