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Development of functional material for deuterium permeation observation under divertor plasma exposures

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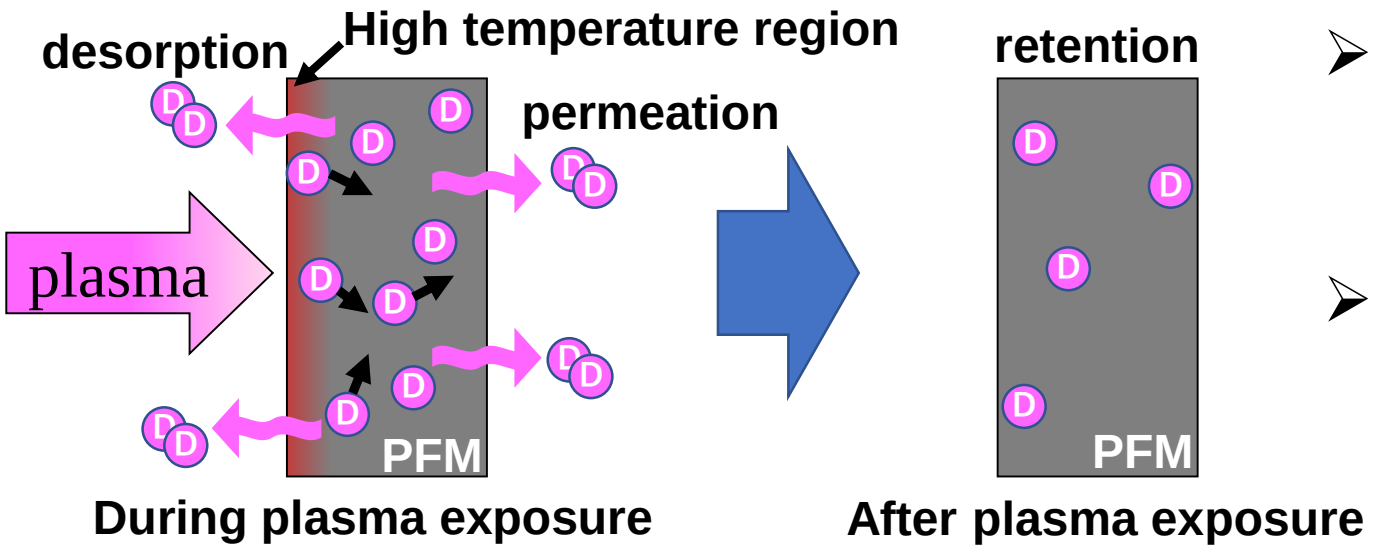
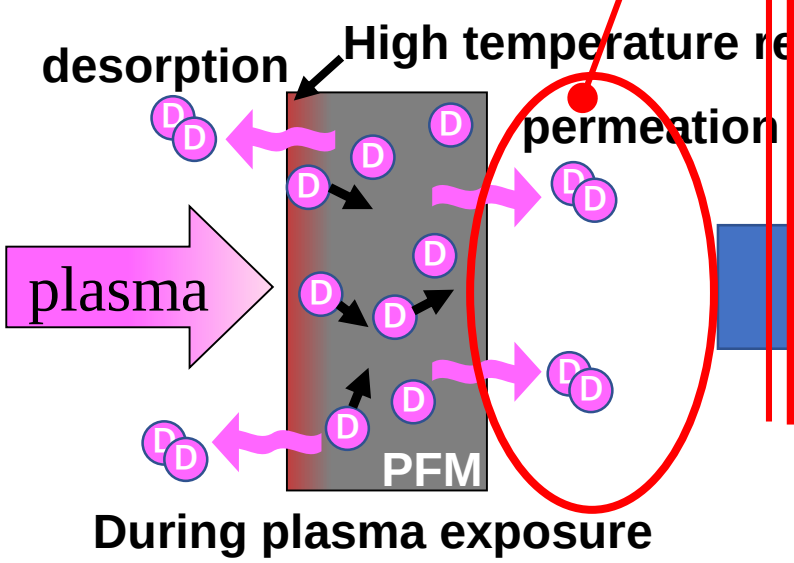
Background

- In DEMO, control of tritium (T) amounts is critical issue because of its radioactivity and necessity of reuse.
- Although hydrogen isotopes diffuse into plasma-facing components (PFCs) under high heat flux and particle flux, its amounts have not been measured.
- Therefore, it is necessary to directly evaluate the retention and permeation amount of hydrogen isotopes in PFCs.



- During plasma exposure, hydrogen isotopes in plasma-facing materials (PFM) are desorbed or permeates.
- After plasma exposure, it can be observed retention of hydrogen isotopes in materials by TDS, and so on.

Background



- **Hydrogen isotope permeation has been investigated in laboratory-scale plasma devices and fusion devices by using quadrupole mass analyzer (QMA) installed behind a membrane.**

  For example: I. Takagi *et al.*, J. Nucl. Mater. **415** (2011) S692-S695.
  H.S. Zhou, Plasma and Fusion Research, 8 (2013) 2402065.

- **However, QMA method is difficult to perform at PFCs under high heat flux and particle flux such as divertor regions due to limited spaces, strong magnetic fields, high heat loads, and so on.**

- **It is required to develop a new method to measure D amounts of permeation in PFCs.**

Motivation & Aim

There is no precedent for direct evaluation of hydrogen permeation in PFCs under high flux plasma exposures in fusion devices.

- **A new method have been proposed that titanium (Ti) and palladium (Pd) are combined with tungsten (W) to store permeated deuterium (D).**
- **It is planed that hydrogen isotopes amount of permeation in PFCs of current fusion devices such as ASDEX-U is measured by this method.**
- **It is expected that characteristics of hydrogen isotopes obtained by the method will contribute to T handling in DEMO.**

In this work, W-Pd-Ti combined samples has been developed and improved in order to use in tokamaks.

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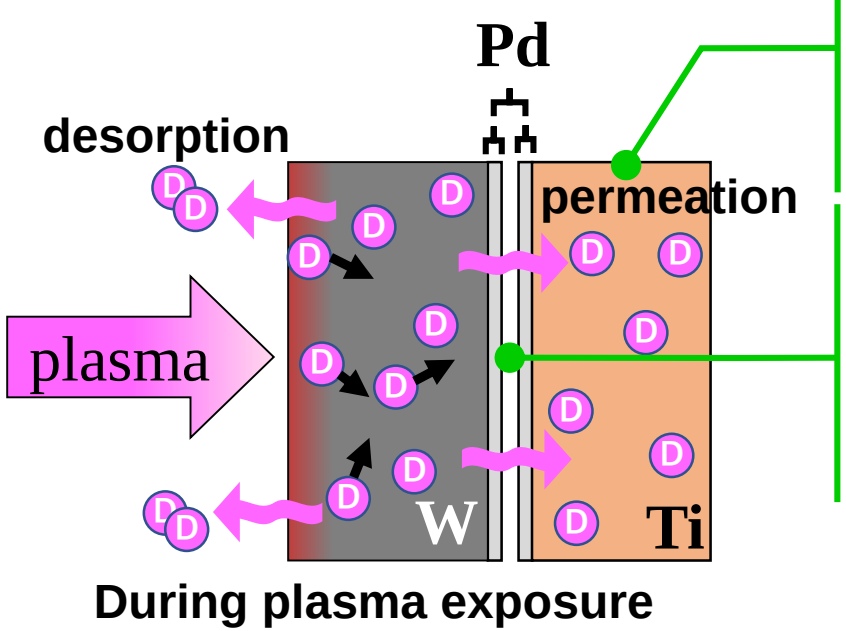
Concept of new method

It has been considered that Ti and Pd are combined with W to store the permeated D.



- In order to store permeated D, we used Ti which is a hydrogen storage metal.
- Ti is installed behind W.

- Pd enhances the D transport from W to Ti.
- Pd is used at contact surfaces of W and Ti as a catalyst.

Concept of new method

It has been considered that Ti and Pd are combined with W to store the permeated D.

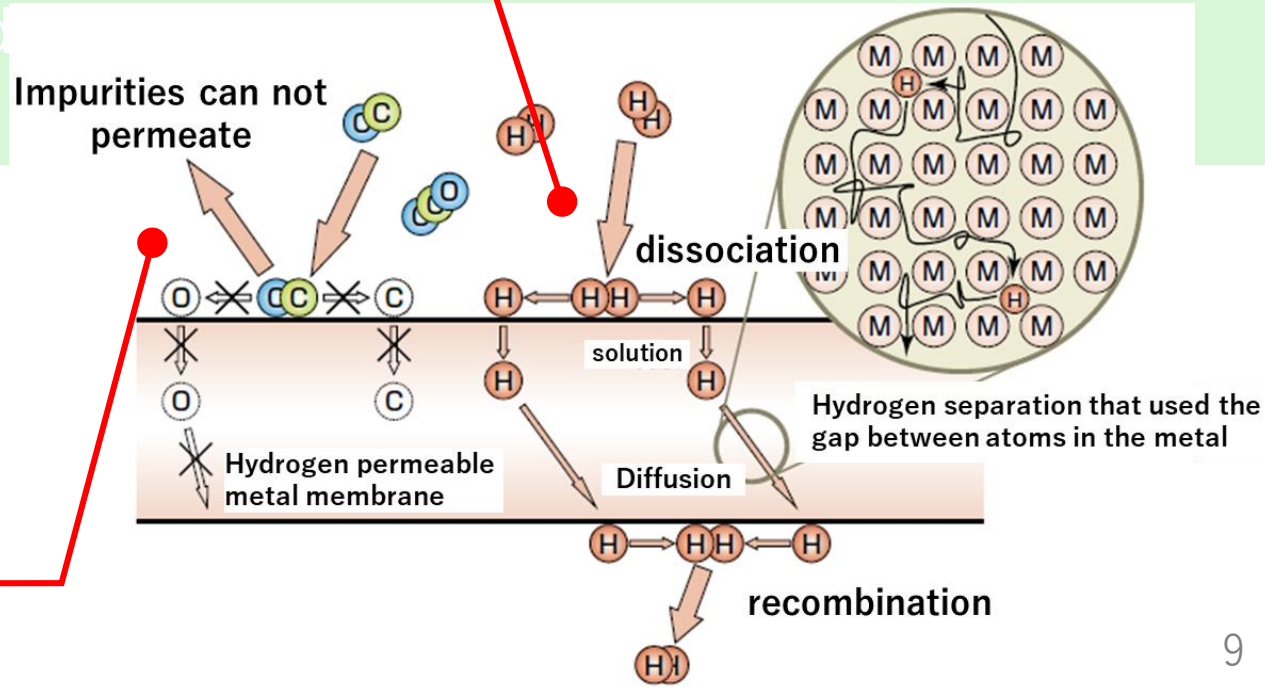
- **Dissociative energies of hydrogen molecules for the diffusion into materials are lower than most of metals.**

For example: E. B. Maxted et al., J. Chem. Soc. 1959, 3130.
H. Nakatsuji et al., J. Am. Chem. Soc. 1985, 107, 26.

- **Due to Pd layer, it is expected that D permeates to base materials as atoms without recombination.**

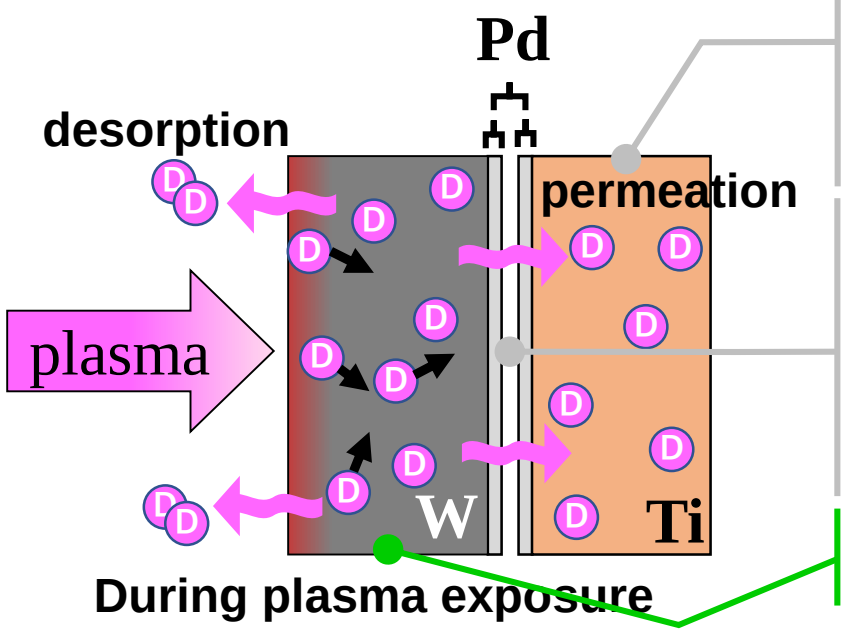
- **Pd is one material of the hydrogen-permselective membranes.**

- **When molecules diffuse into materials, they must be dissociated at the surface.**
- **A dissociation needs high energy.**



Concept of new method

It has been considered that Ti and Pd are combined with W to store the permeated D.



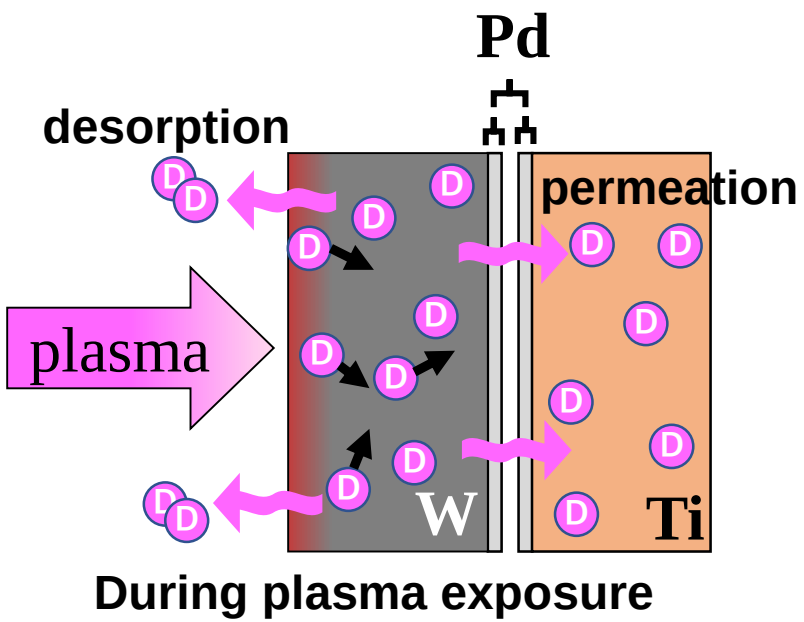
- In order to store permeated D, we used Ti which is a hydrogen storage metal.
- Ti is installed behind W.

- Pd enhances the D transport from W to Ti.
- Pd is used at contact surfaces of W and Ti as a catalyst.

- **W is a candidate of PFMs.**

Concept of new method

It has been considered that Ti and Pd are combined with W to store the permeated D.



During plasma exposure

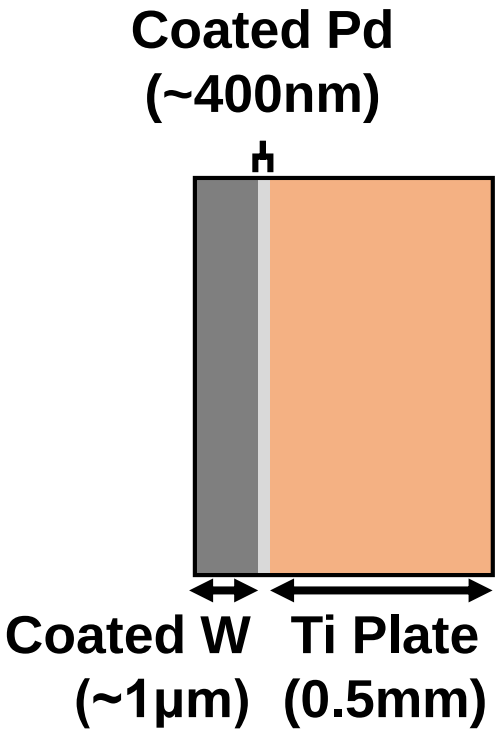
- From these reasons, the combination of the materials was selected.
- In the previous study, D amounts of permeation had been observed as D retention in Ti. [1]
- In these samples [1], Ti and W were separating, and the contact between W and Ti via Pd was as only touching.

[1] T. Hayashi, T. Takimoto, *et.al.*, FED, **136** (2018) 545.

However, in order to experiments in tokamaks, it is necessary to modify the samples from separating structure.

In this work, integrated-type samples have been newly developed.

Design of integrated samples



Ti plate (15 mm x 15 mm) has been applied mirror polish.

Magnetron sputtering

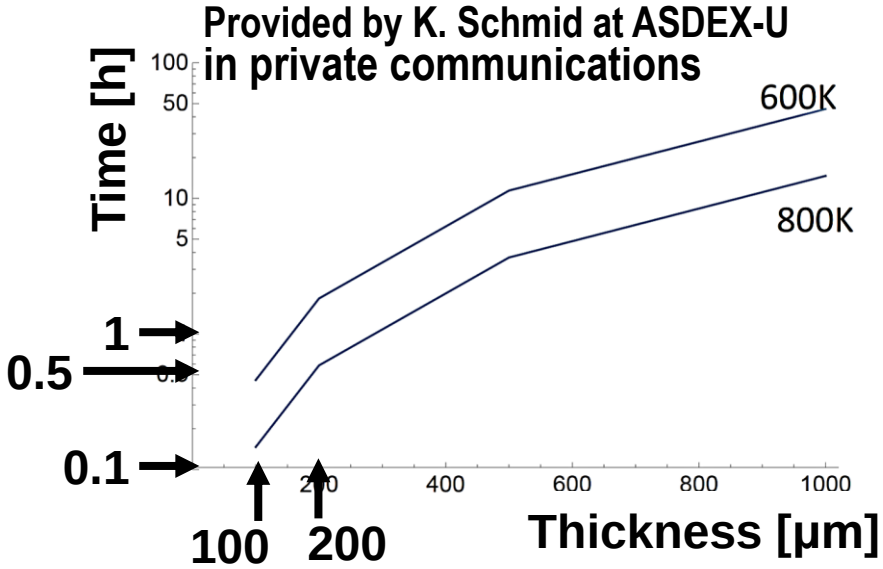
Pd has been coated (~400 nm) on Ti plate

Magnetron sputtering

W has been coated (~1 μm) on Pd layer

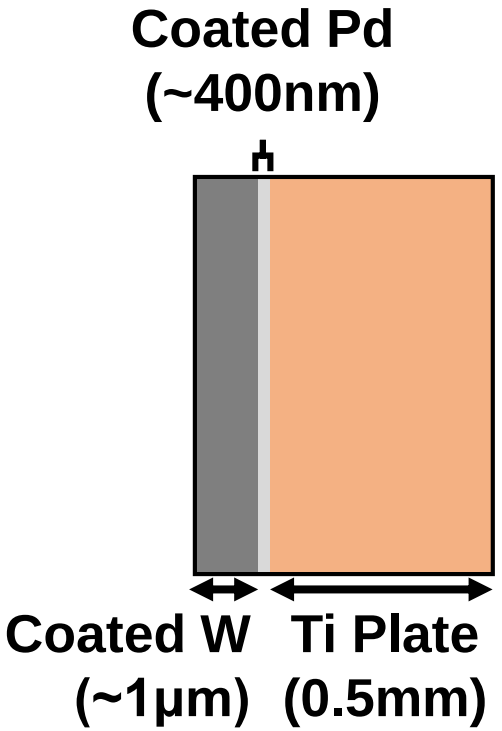
Estimate permeation break through time

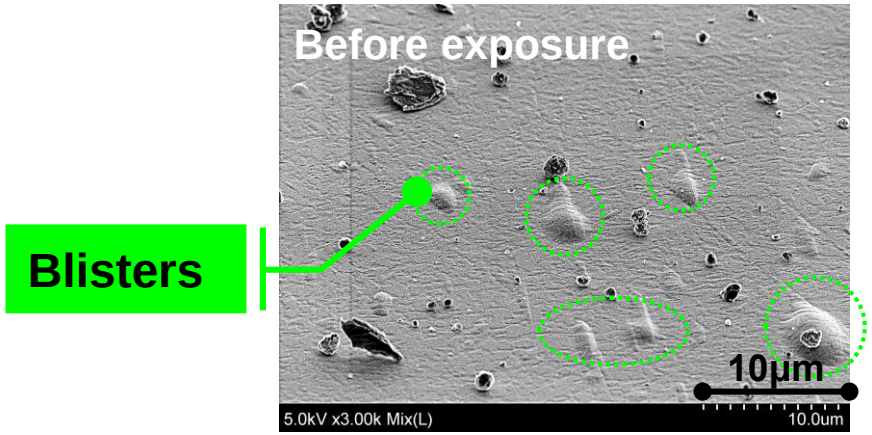
Extrapolation to AUG @ 600K vs 800K



- Comparing D permeation time through W and a pulse length of discharges of ASDEX-U (<0.01h), W thickness must be less than 100μm at least.
- In order to produce such thin W layer, coated W was selected.

Design of integrated samples

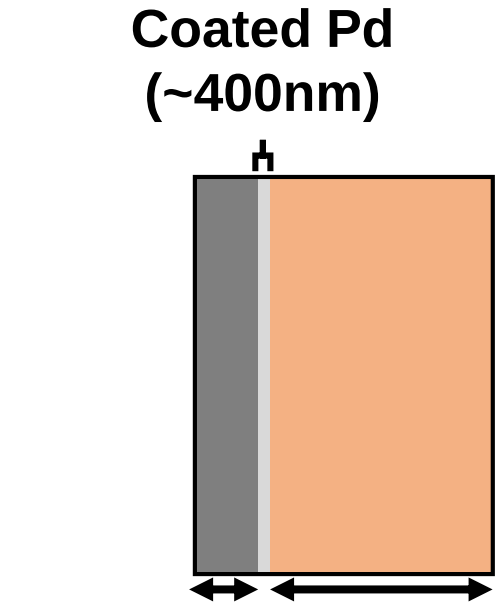


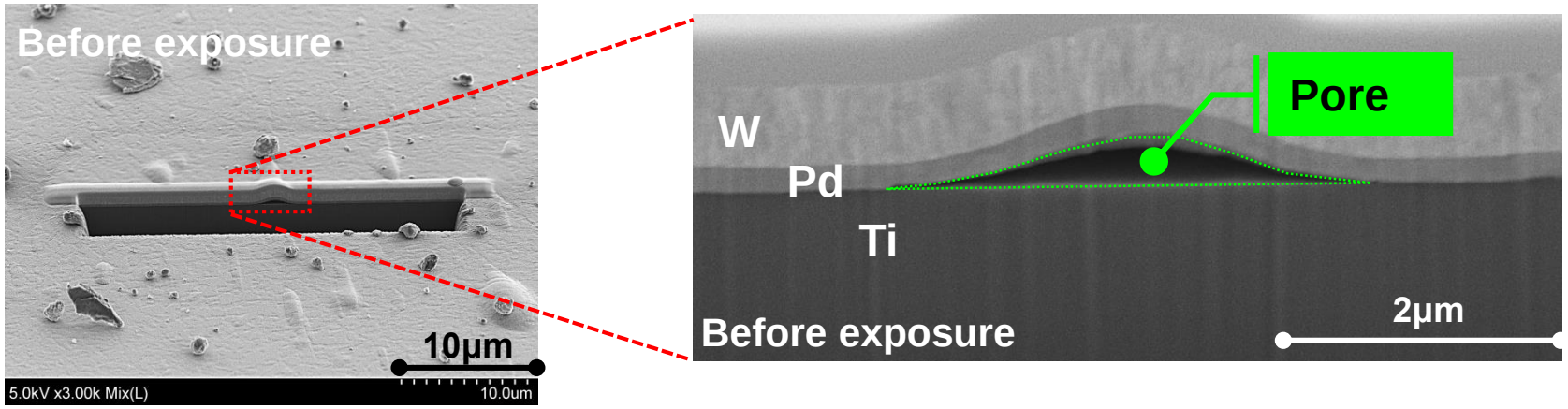


Surface morphology by Scanning Electron Microscope (SEM).

- Surface observations were performed on produced W-Pd-Ti samples
  - Blisters (several µm) were observed on the surface.

Design of integrated samples



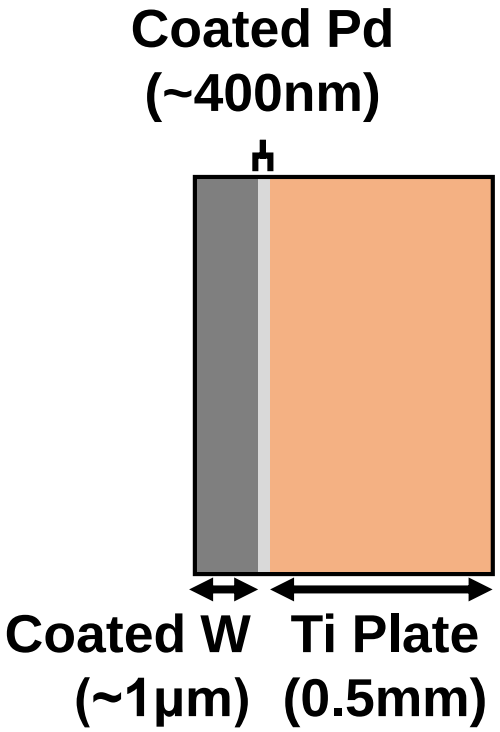


Cross-sectional images after Focused Ion Beam (FIB).

- Surface observations were performed on produced W-Pd-Ti samples
  - Blisters (several μm) were observed on the surface.
- Cross-sectional observations were performed on same samples.
  - Pores (width of several μm) were observed around the boundary between Pd and Ti.
  - It is not satisfied a good contact between Pd and Ti required to smoothly permeation due to Pd.

It is necessary to enhance the contact between Pd and Ti.

Preparation of samples



Ti plates (15 mm x 15 mm) have been applied mirror polish

Magnetron sputtering

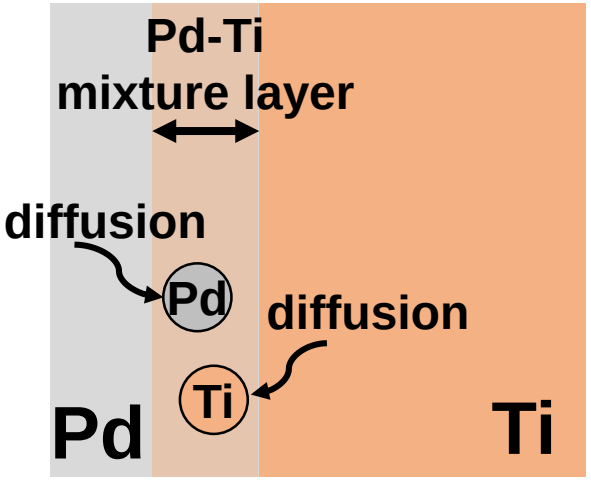
Pd has been coated (~400 nm) on Ti plates

Infrared heating

Heat treatment to Pd-Ti samples has been performed

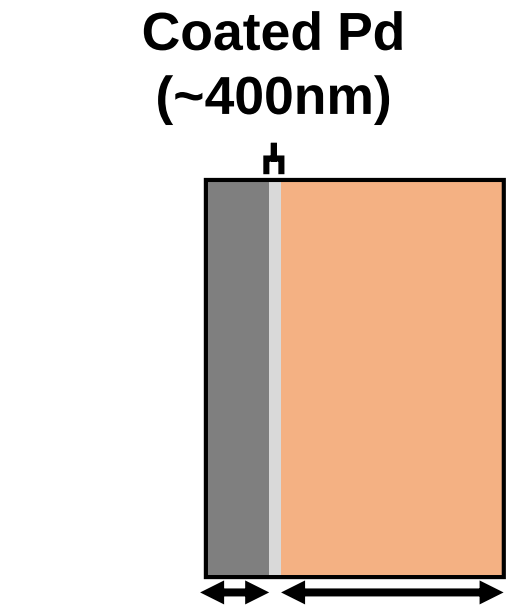
Magnetron sputtering

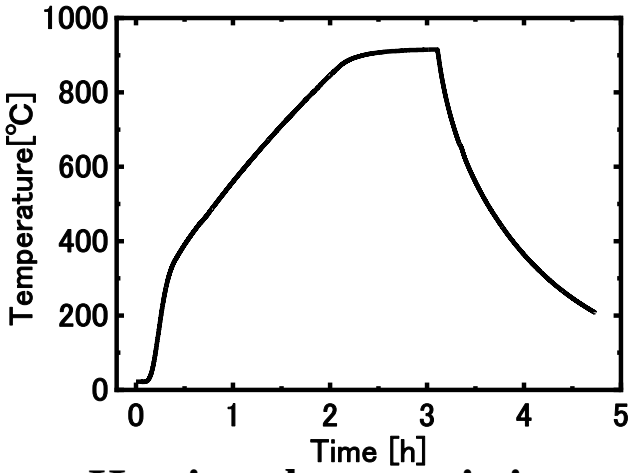
W has been coated (~1 μm) on Pd layer



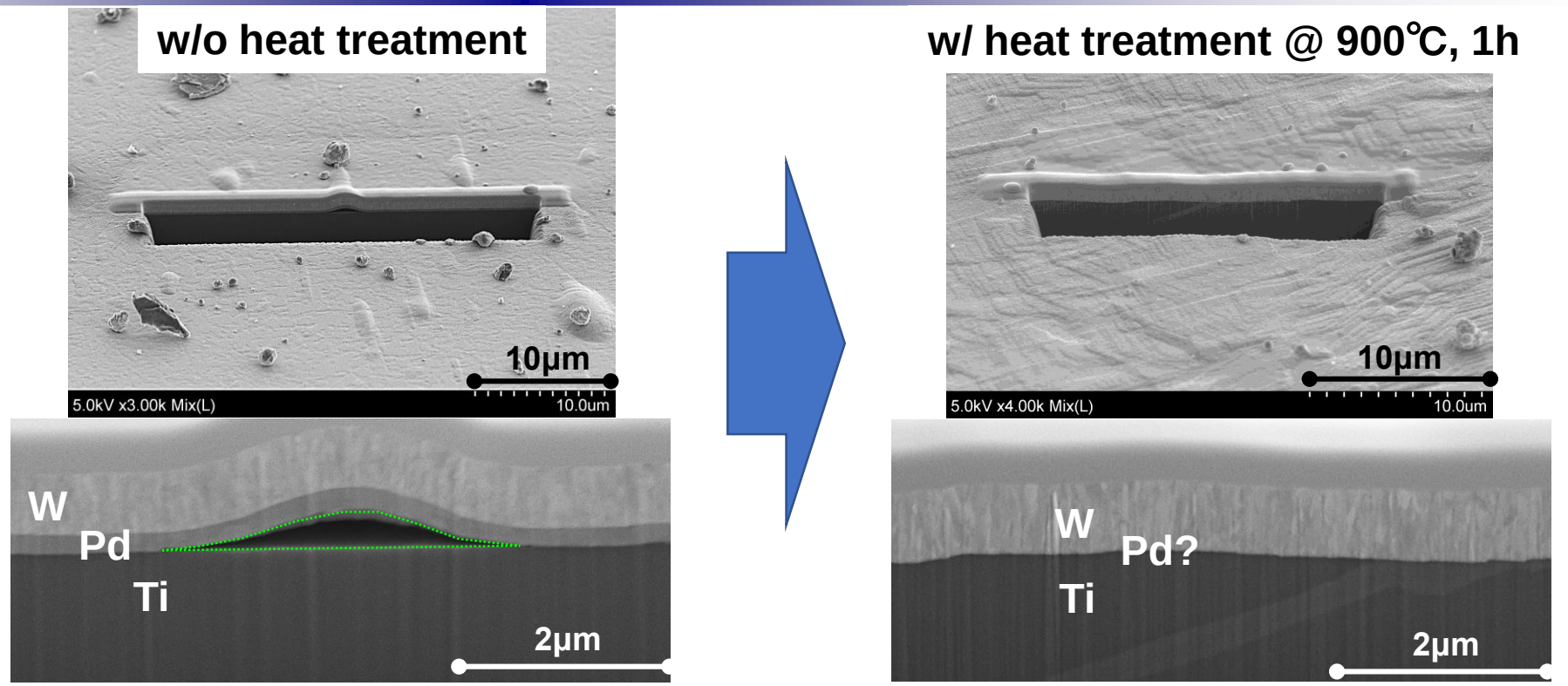
Heat treatments have been performed to enhance the contact between these layers by promoting the diffusion of metal particles into each other layer.

Preparation of samples





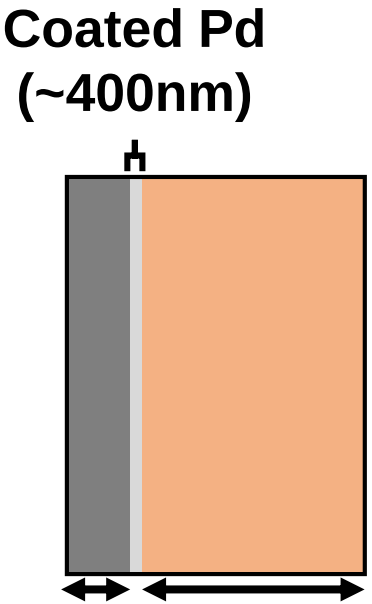
Heating characteristic

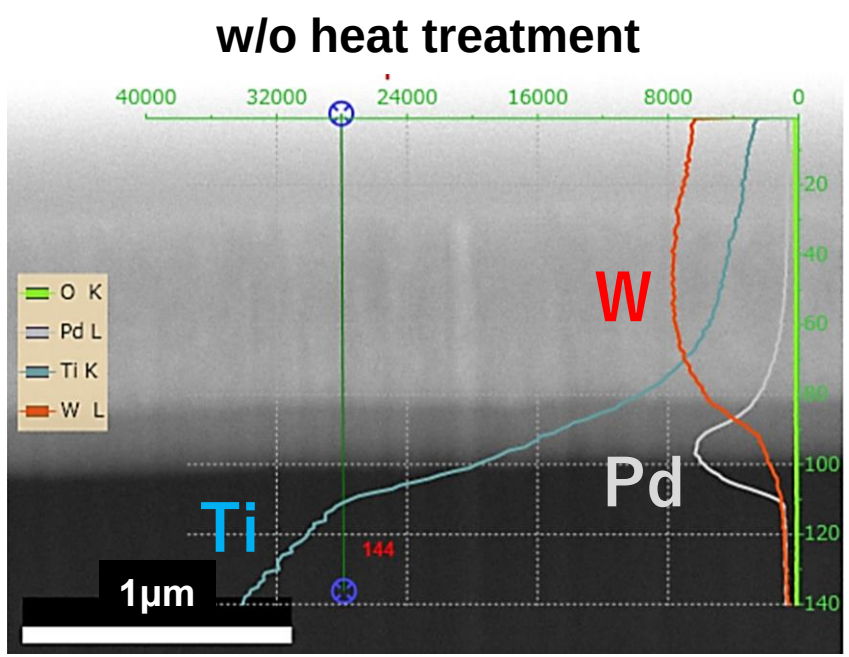


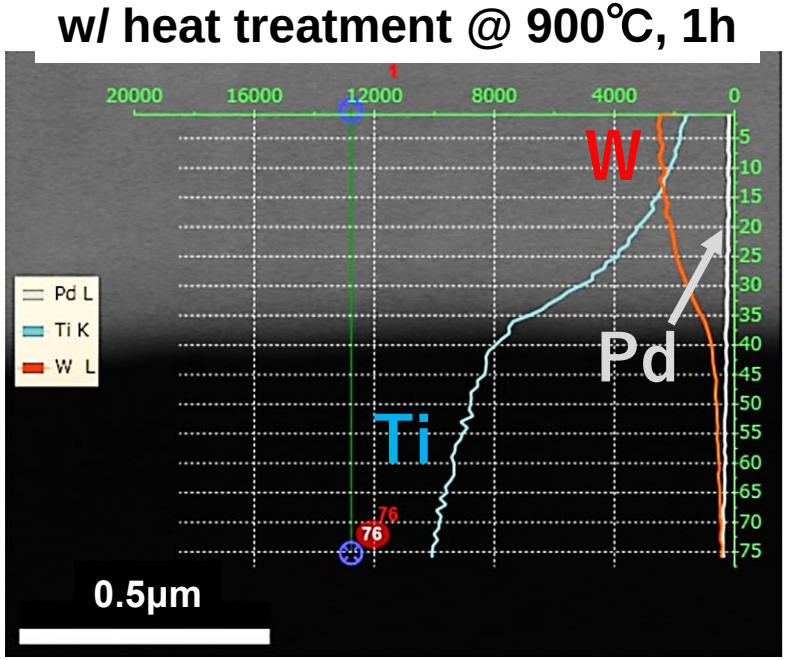
Cross-sectional images after FIB.

- Surface and cross-sectional observations were performed on also W-Pd-Ti sample with a heat treatment.
  - After heat treatment, blisters were not observed on the surface.
  - Also, pores were not observed between W and Ti after heat treatment.

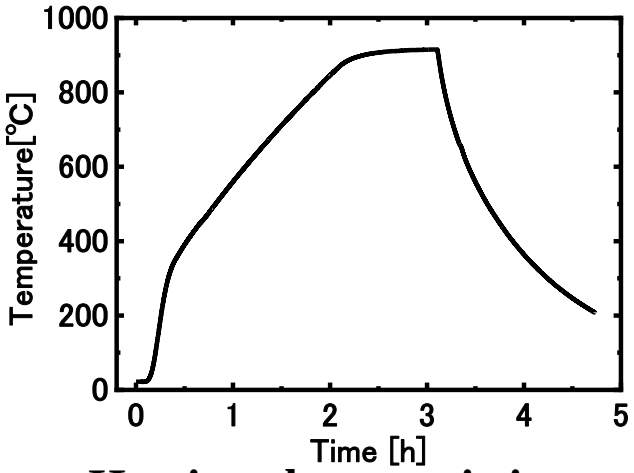
Preparation of samples







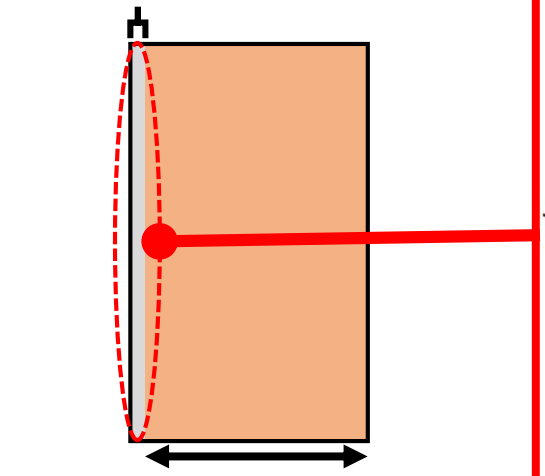
The results of Energy dispersive X-ray spectrometry (EDX) on the cross-section of samples.

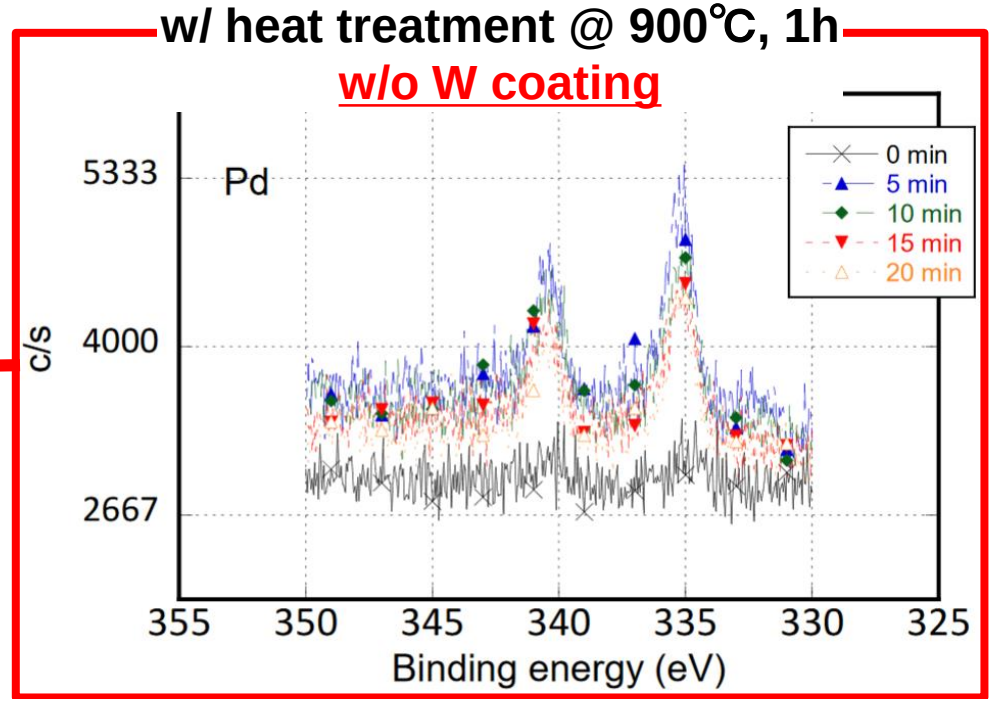


Heating characteristic

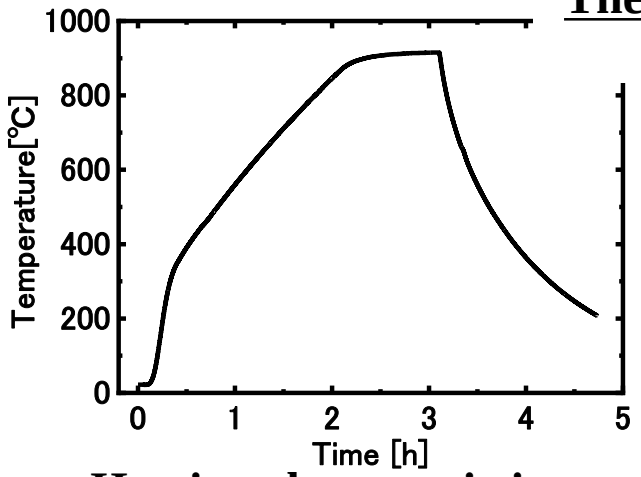
- EDX analysis was performed on the cross-sections of both samples.
  - After a heat treatment, Pd layer was not clear observing by using EDX.

Preparation of samples



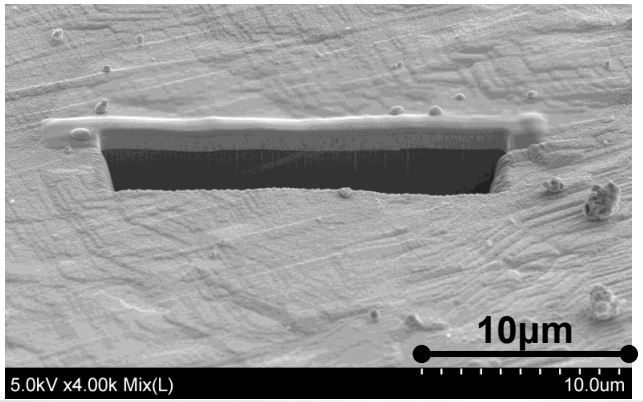


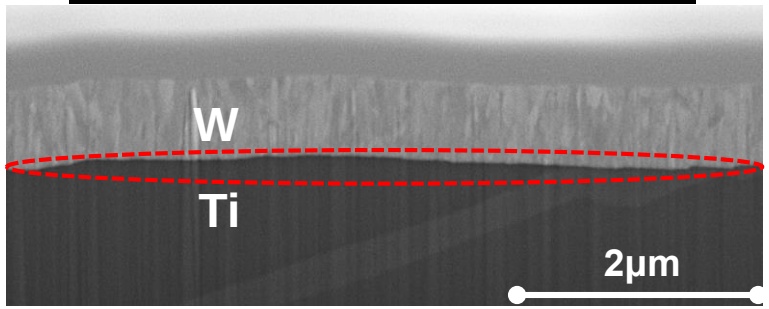
The results of X-ray Photoelectron Spectroscopy (XPS) on the surface of the Pd-Ti sample.



Heating characteristic

w/ heat treatment @ 900℃, 1h

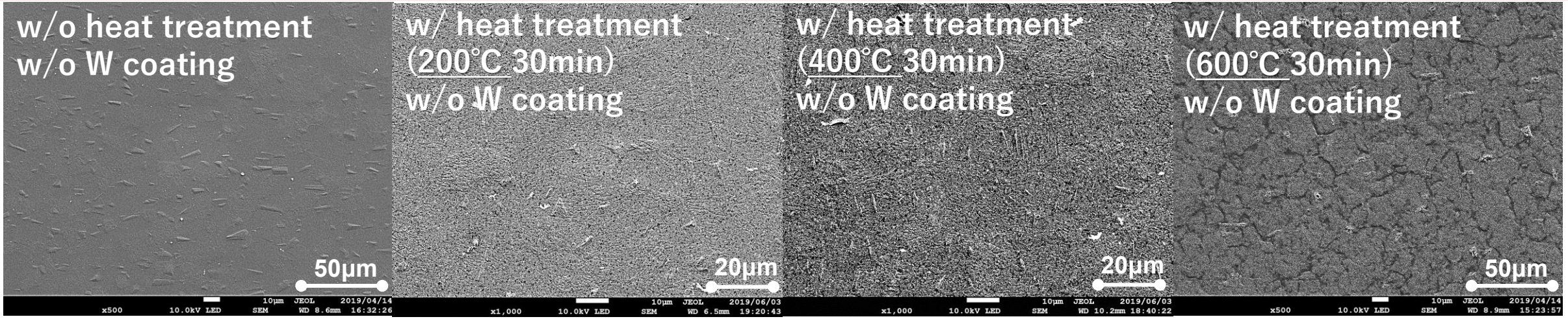




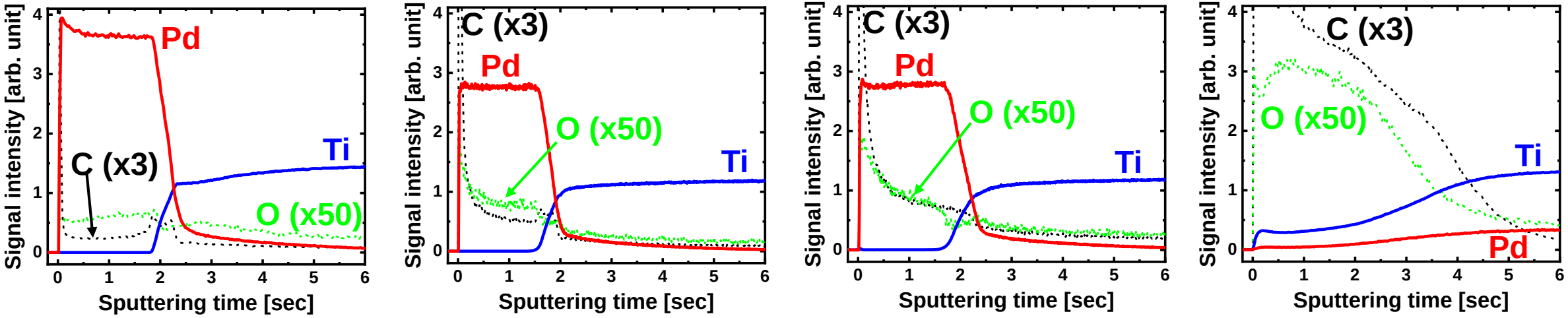
- XPS analysis of the surface was performed on the Pd-Ti sample without W coating after same heat treatment.
  - Pd layer was confirmed on the surface of the Pd-Ti sample after the heat treatment.
  - Pd layer remains on Ti plate after the heat treatment.

Preparation of samples

It has been clarified that due to heat treatments for 30min at 400℃ or less, pores between Pd and Ti are removed and the Pd layer is also remained.



Surface morphologies by SEM on each Pd-Ti sample which varied temperature of heat treatment.



Depth profiles of elements in each Pd-Ti sample by Glow discharge optical emission spectrometry (GD-OES).

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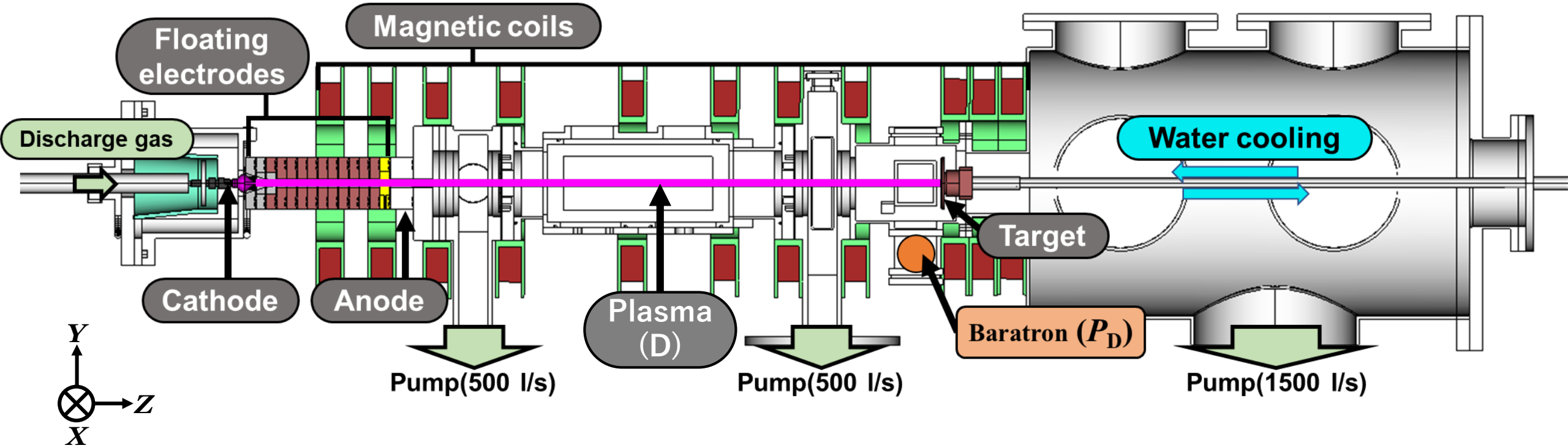
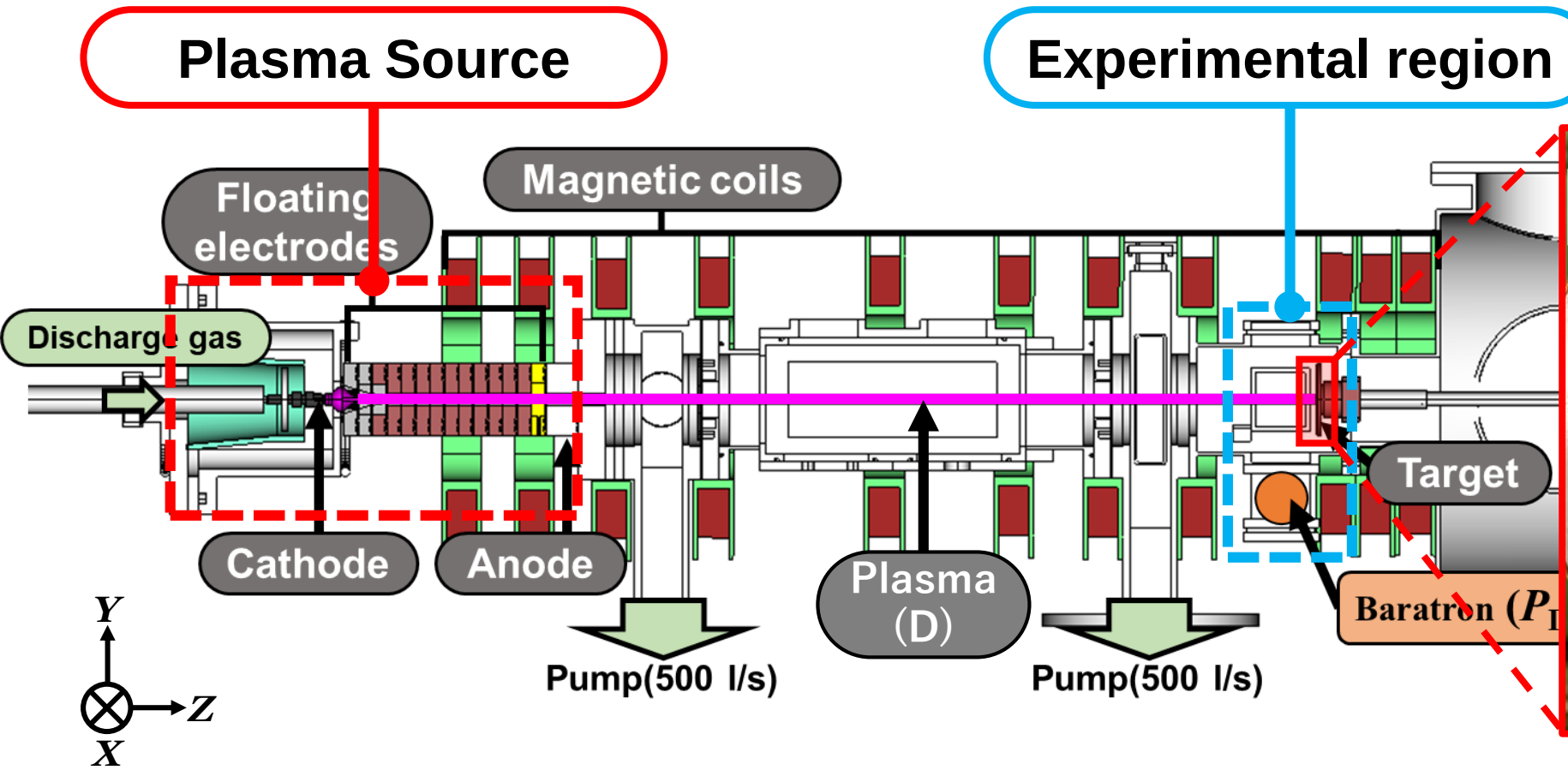
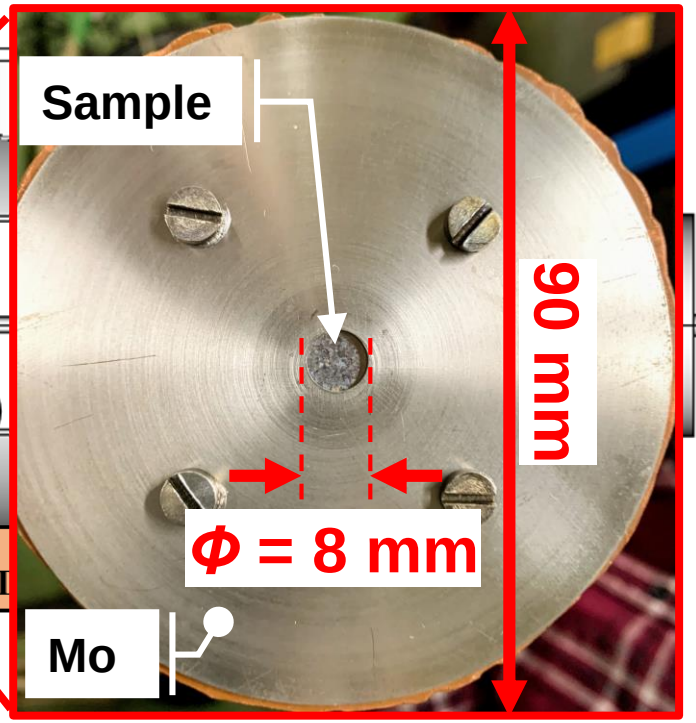
Linear Plasma Device TPDsheet-U @ Tokai Univ.

Plasma exposures have been performed to W-Pd-Ti samples using linear plasma device TPDsheet-U.



Linear Plasma Device TPDsheet-U @ Tokai Univ.

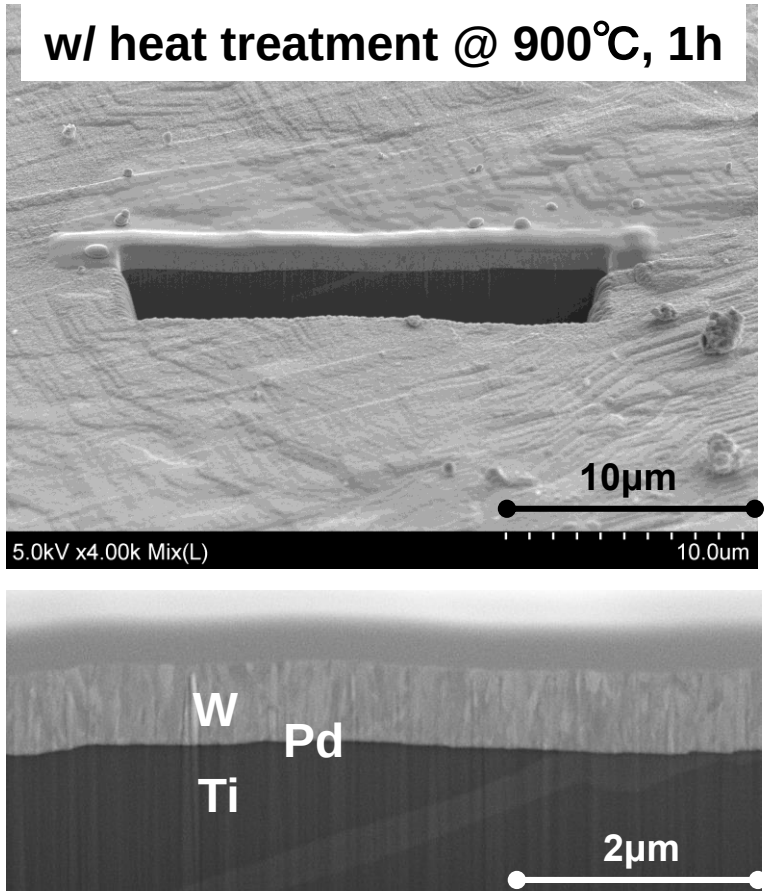




- This target is floating electrically.
- Target potential was around -15V during plasma exposures.

Experimental Conditions

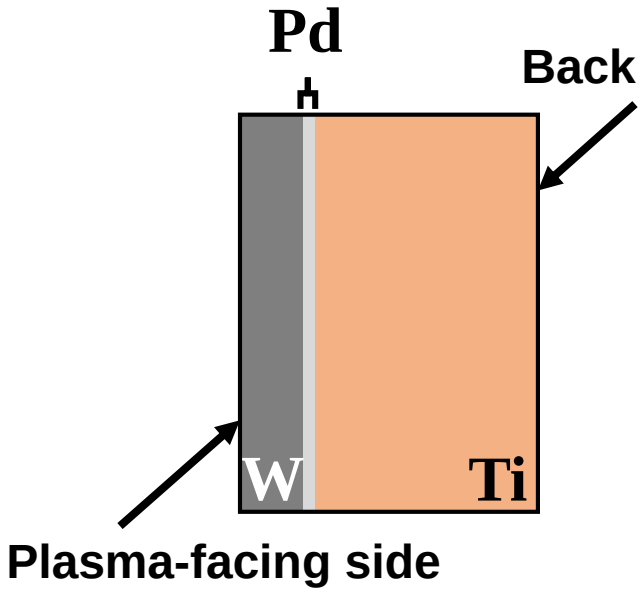
- **The plasma exposures have performed to the W-Pd-Ti samples which Pd layer is thin due to the heat treatment for 1h at 900℃ by using TPDsheet-U.**

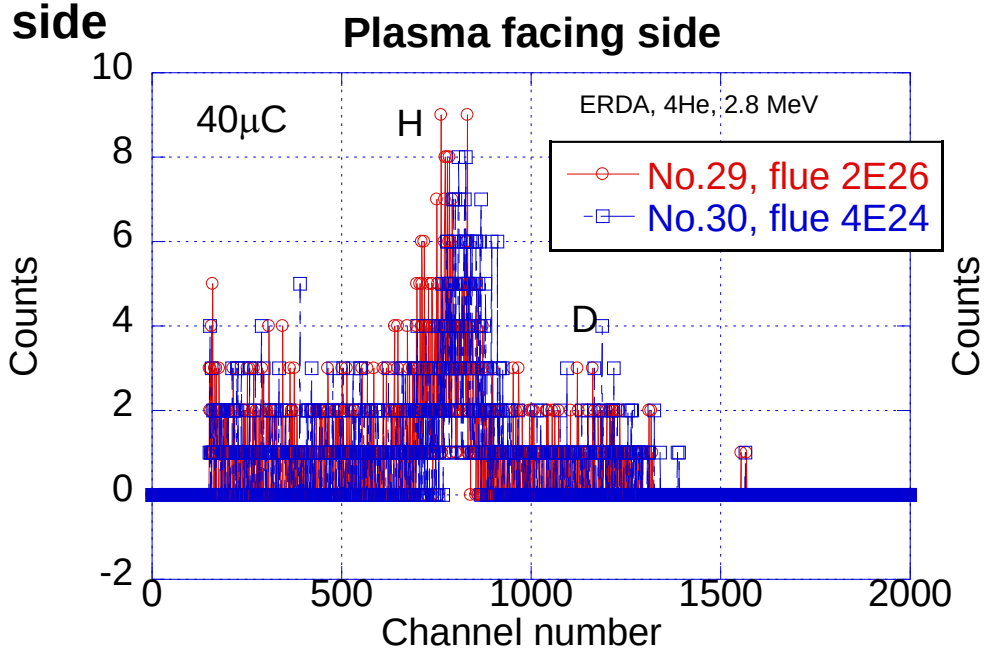


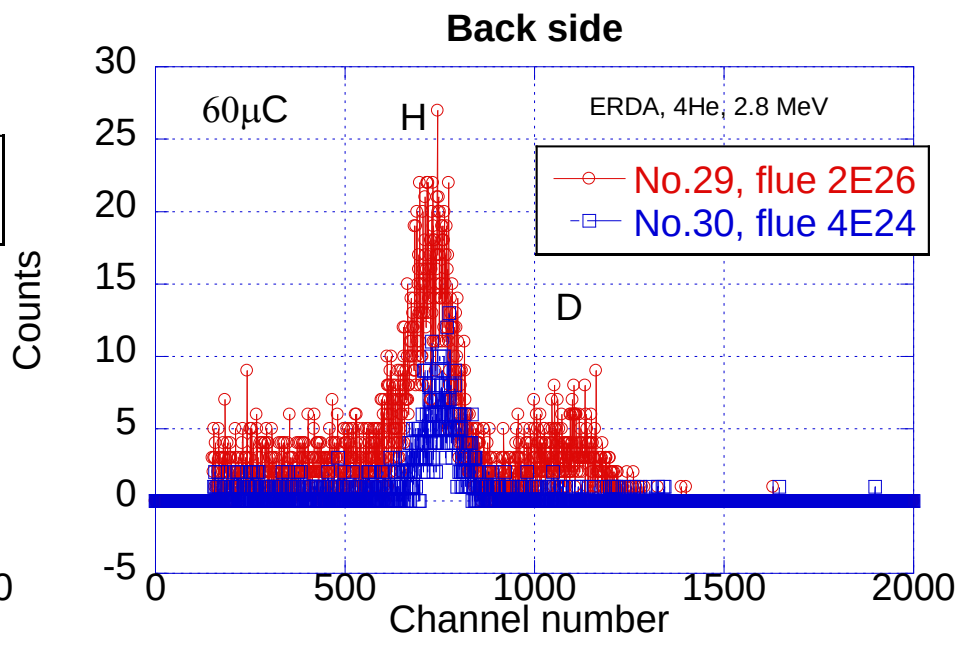
Plasma Conditions

Sample No.	#29	#30
Ion Flux Γ_i [$m^{-2}s^{-1}$]	~ 1.8×10^{22}	
Electron Density n_e [m^{-3}]	~ 7.7×10^{17}	
Electron Temp. T_e [eV]	~ 4.8	
Exposure Time t	3 h	4 min
Ion Fluence $\Gamma_i \cdot t$ [m^{-2}]	~ 2.0×10^{26}	~ 4.5×10^{24}
	High fluence	Low fluence

Results of plasma exposures







Spectrum of Elastic Recoil Detection Analysis (ERDA) .

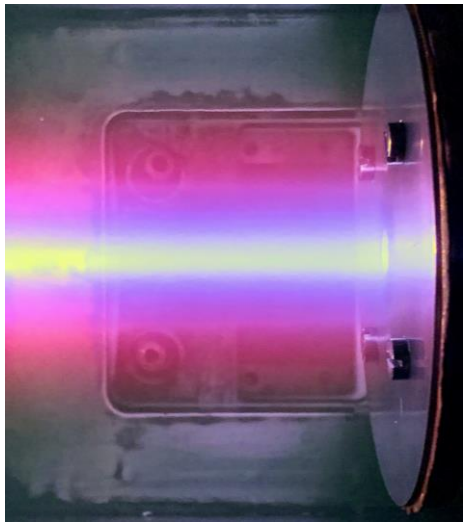


Photo of the experiment

- **Measurements of D amounts by ERDA were performed on sample surfaces of plasma-facing side and back side.**
  - **D was detected on both sides of samples.**
  - **At the back side, D was detected in only high fluence condition (#29).**

It has been success to detect D on Ti surface (back side) of W-Pd-Ti sample.

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Summary & Future Plans

Summary

- ✓ There is no precedent for direct evaluation of hydrogen permeation in PFCs under high flux plasma exposures in fusion devices.
  - We have newly developed the integrated W-Pd-Ti sample as a new method to quantitatively evaluate the amount of hydrogen permeation during high flux plasma exposures in fusion devices.
- ✓ In first sample preparation, pores which it is considered to impede D permeation to Ti were observed around the boundary between Pd and Ti layers.
  - It has been clarified that due to heat treatments for 30min at 400℃ or less, pores between Pd and Ti are removed and the Pd layer is also remained.
- ✓ Plasma exposures have been performed to W-Pd-Ti samples using TPDsheet-U.
  - It has been success to detect permeated D at Ti surface (back side of samples) by ERDA after plasma exposure with enough ion fluence (ex. ~2.0E26 m^{-2}).

Future Plans

- ☐ Investigation of fluence dependence and material, which is W-Pd-Ti samples, temperature dependence of plasma driven permeation in TPDsheet-U.
- ☐ Plasma exposures in fusion devices such as ASDEX-U.

Thank you for your attention

Acknowledgement

This work is performed with the support and under the auspices of the NIFS Collaboration Research program (NIFS17KEMF109).