8th International Workshop on Anomalies in Hydrogen/Deuterium Loaded Metals, Oct13-18, 2007,Catania, Italy

Changes in Surface Layer Impurities of Pd Due to Heat and/or *Deuterium*-Permeation Treatments and Their Influence on *Deuterium* Permeability

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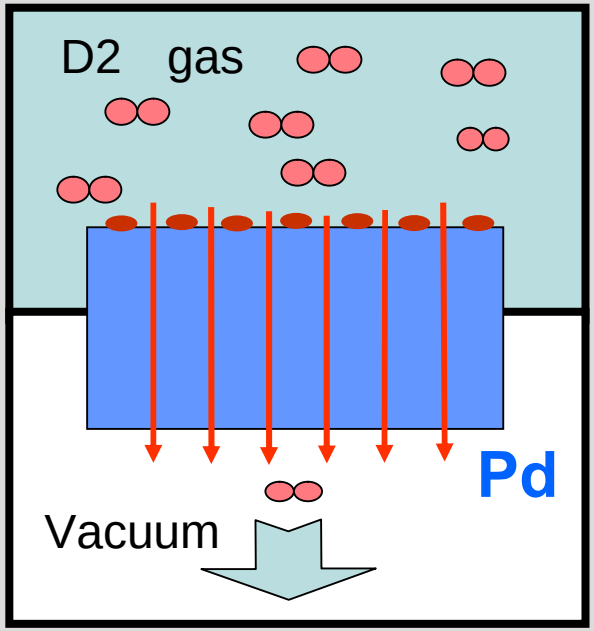
Outline

1. Introduction
2. Change of elemental concentration on Pd *by heat treatment*
   - Annealing in vacuum, in air
   - Sulfur and Oxygen
3. Change of elemental concentration on Pd *with D_2 permeation*
   - Sulfur and Oxygen
4. Improving the rate of D_2 permeation through Pd by heat treatment

D2 Permeation through Pd

Iwamura et al

(Jpn.J.Appl. Phys.41(2002)4642)

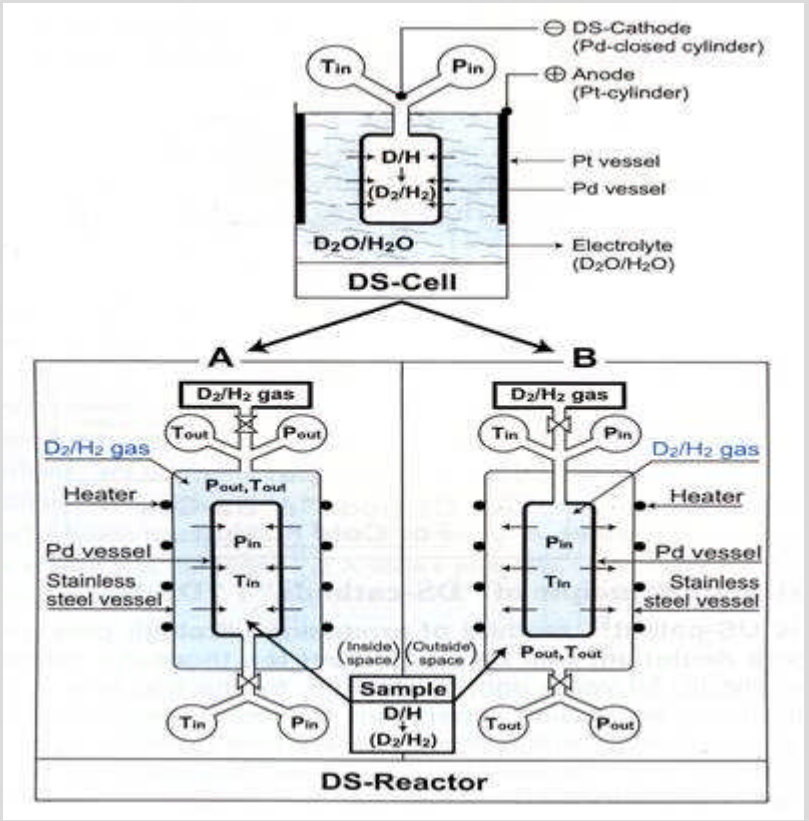


Nuclear Transmutation

$$^{88}Sr \rightarrow {}^{96}Mo$$

$$^{133}Cs \rightarrow {}^{141}Pr$$

Y.Arata and Y.Zhang (ICCF12)



⇒"Good efficiency "cold fusion

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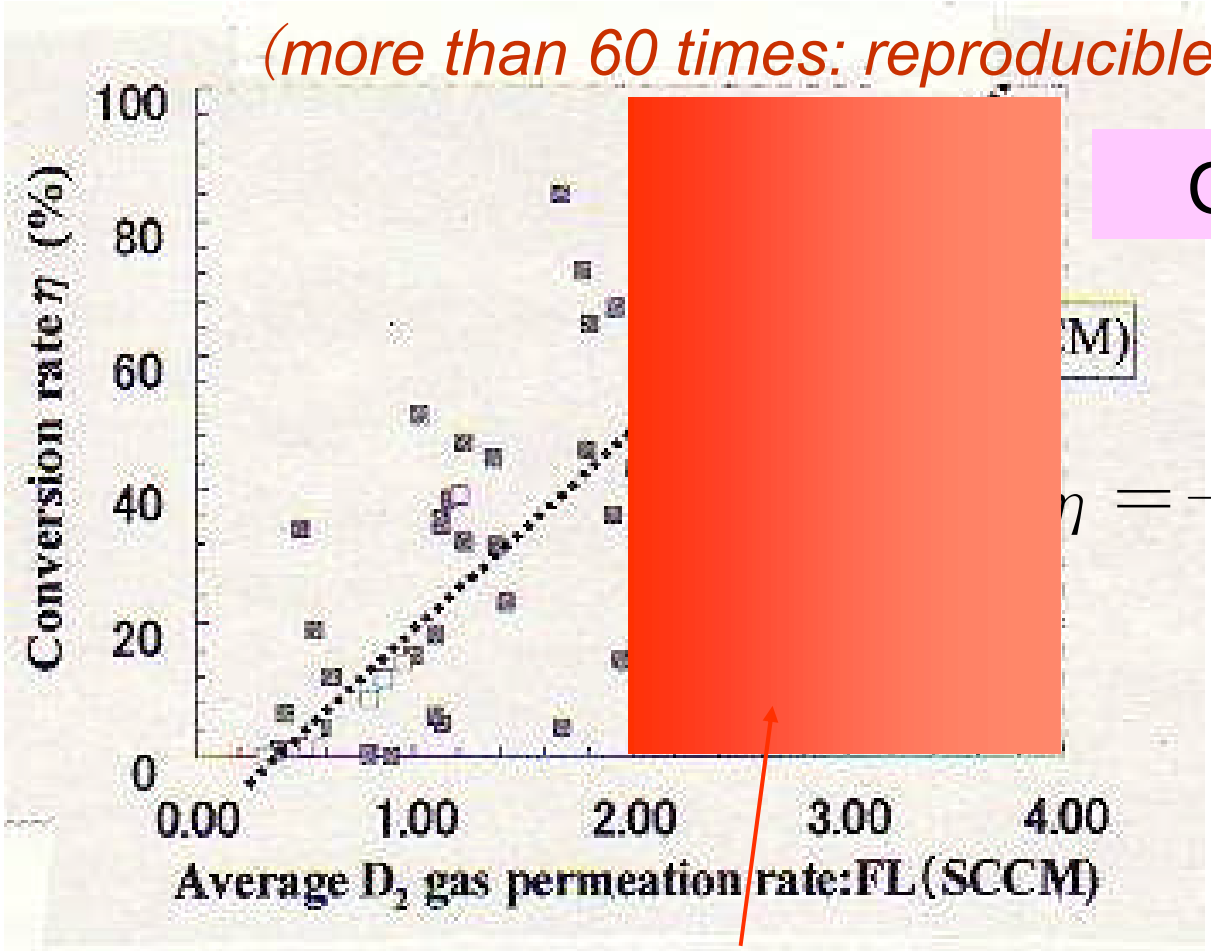
Conversion Rate vs D2 Permeation Rate

Iwamura et al. (2003, iccf10)

(more than 60 times: reproducible)



Cs →Pr

$$\eta = \frac{[Pr]}{[Pr] + [Cs]}$$

High permeation rate is desired !

(larger than 2 SCCM)

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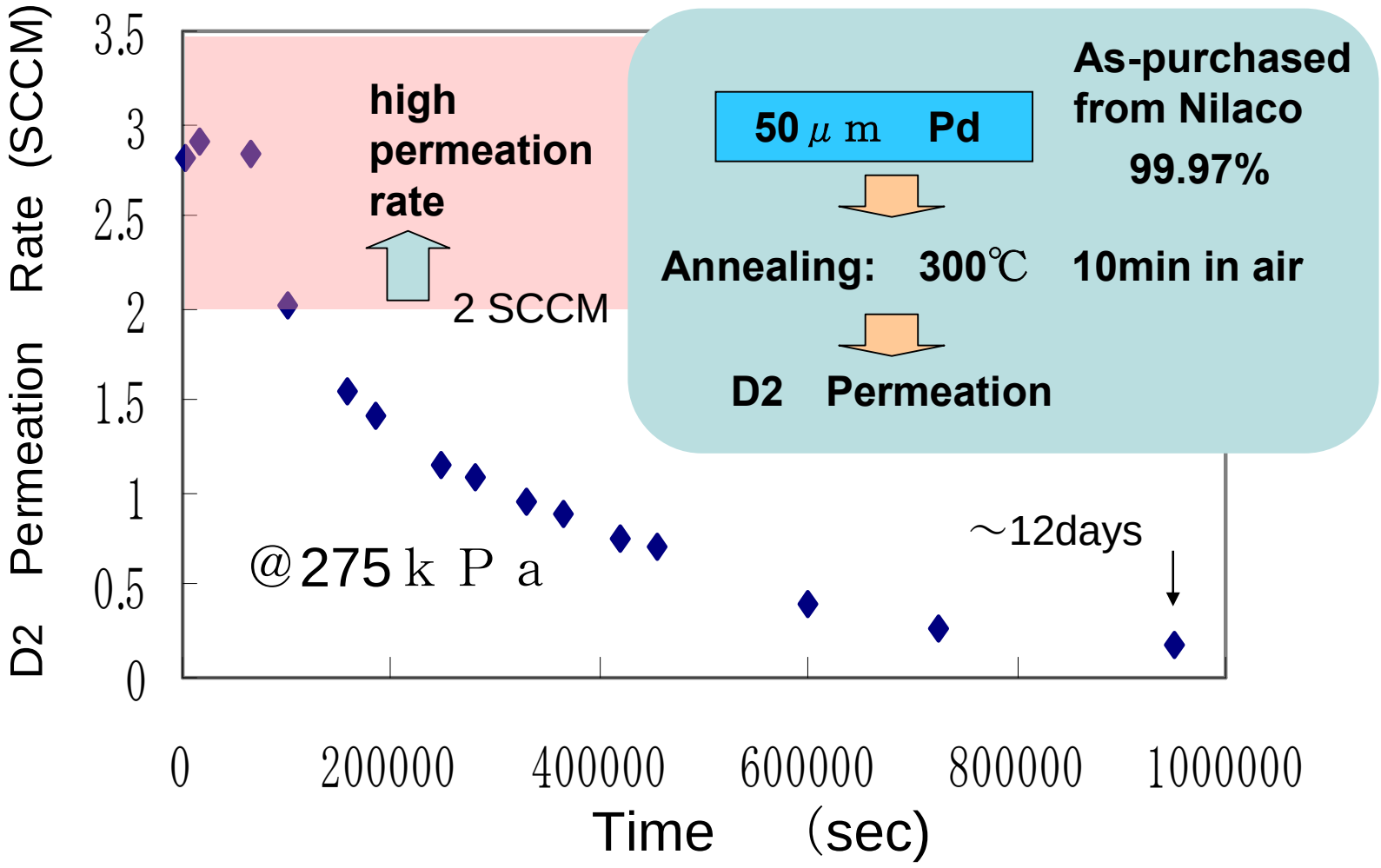
Example of D2 permeation rate vs time



(＊SCCM: Standard Cubic Centimeter per Minute)

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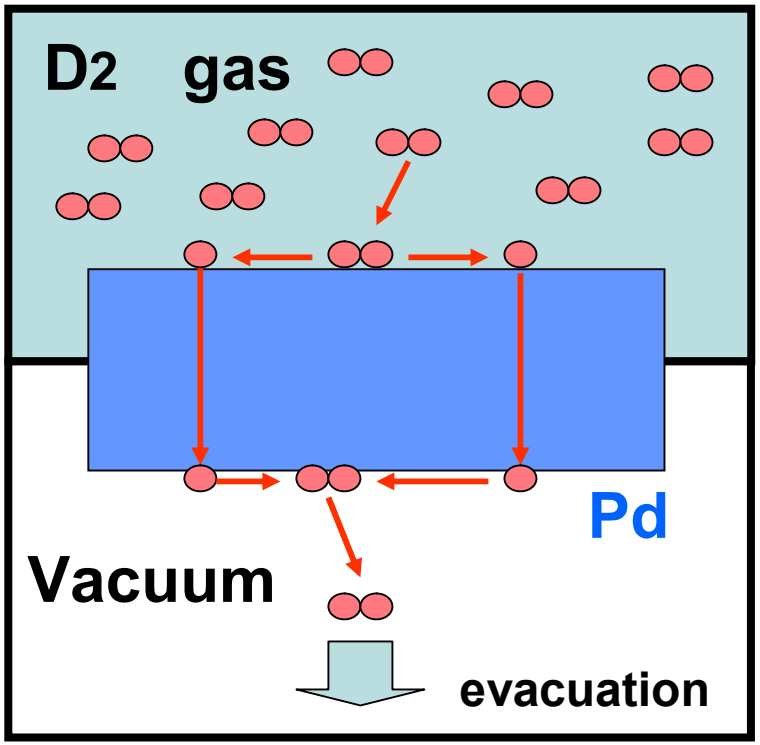
D2 Permeation through Pd



For high permeation rate

Dissociative Adsorption
(front surface)
&
Recombination (back surface)
are to be active

Both surfaces are to be clean

Pd is annealed in air at ≧300℃ as a pretreatment to remove organic carbon

Annealing condition before D2 Permeation

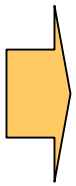
Pd foil

Nilaco 50 μm

99.97%

major impurities
(by supplier)

Pt	210 ppm
Ru	24 ppm
Ag	21ppm
Rh	21ppm
Fe	11ppm
Si	11ppm



Annealing Condition

- in air 500℃ 10min
- in air 1000℃ 10min
- in air 950℃ 2hr
- in vacuum 950℃ 2hr

D2 permeation

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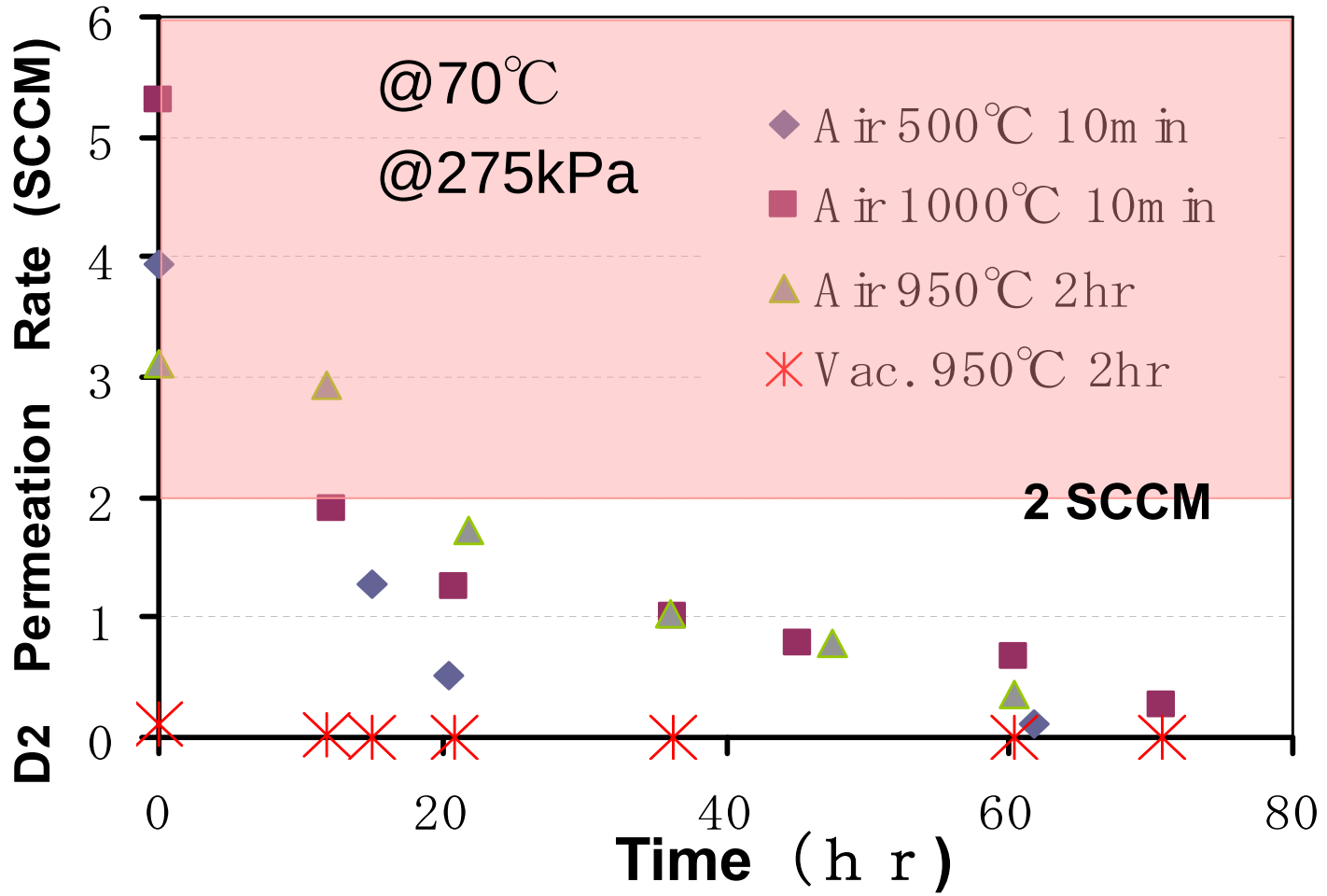
Effect of heat treatment on D2 permeation



- Vacuum annealed sample : almost no permeation
- Air annealed sample :initially high but degrades quickly

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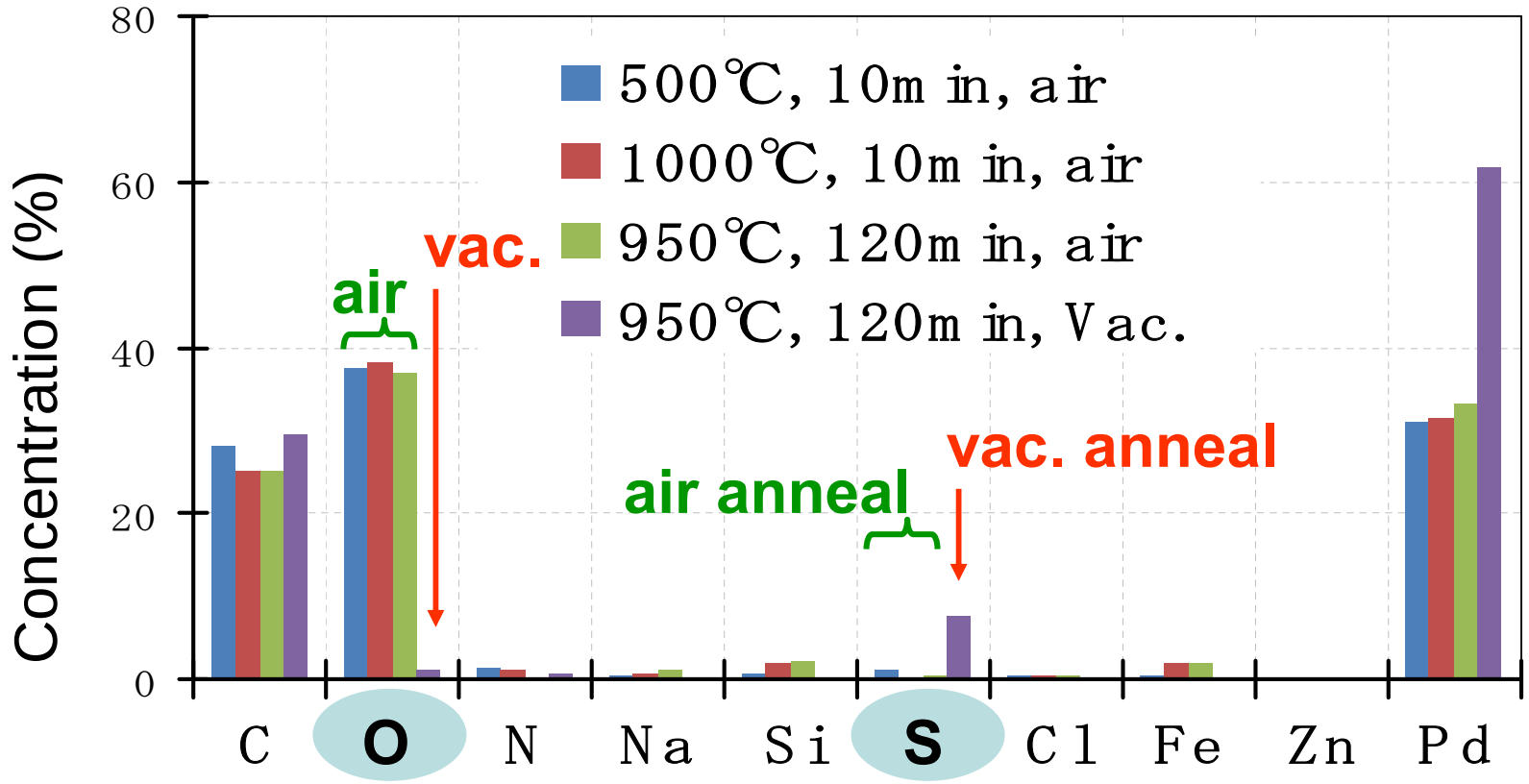
Elemental concentration on the surface of Pd *after heat treatments* (by XPS)



Vac anneal ⇒ high S concentration at the surface

Air anneal ⇒ low S concentration ⇒initial high permeability

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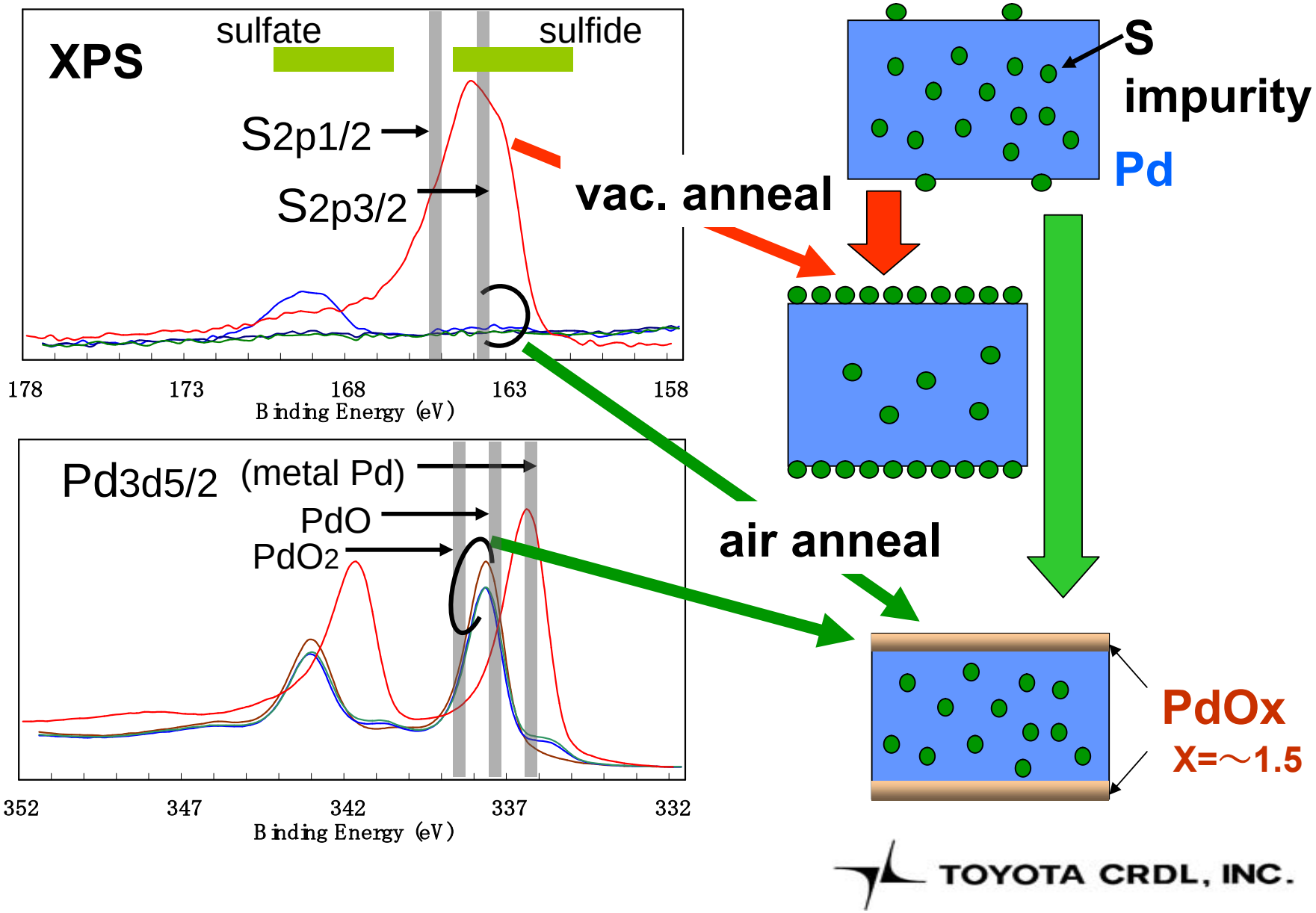
Alteration of Pd surface by annealing



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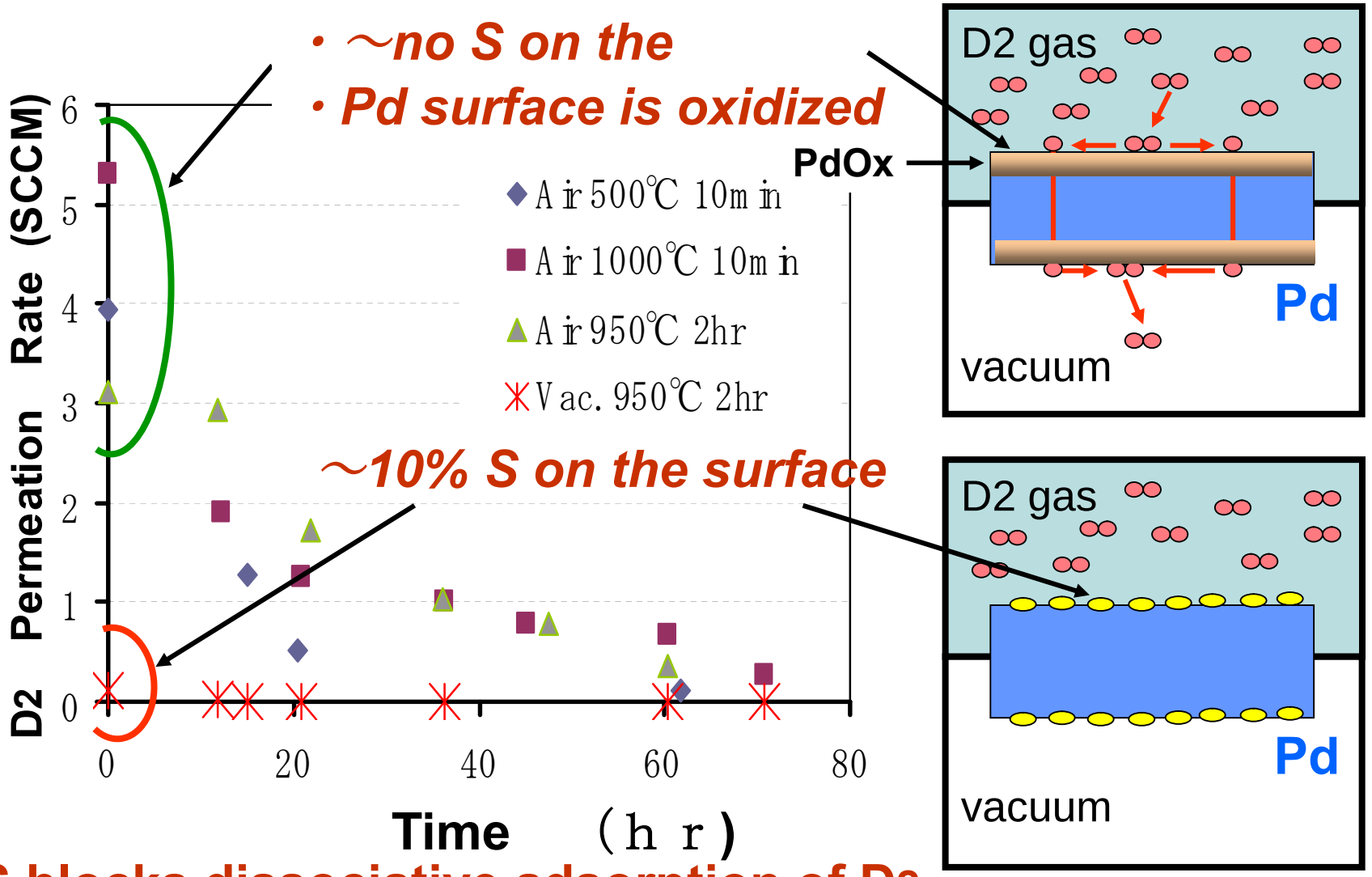
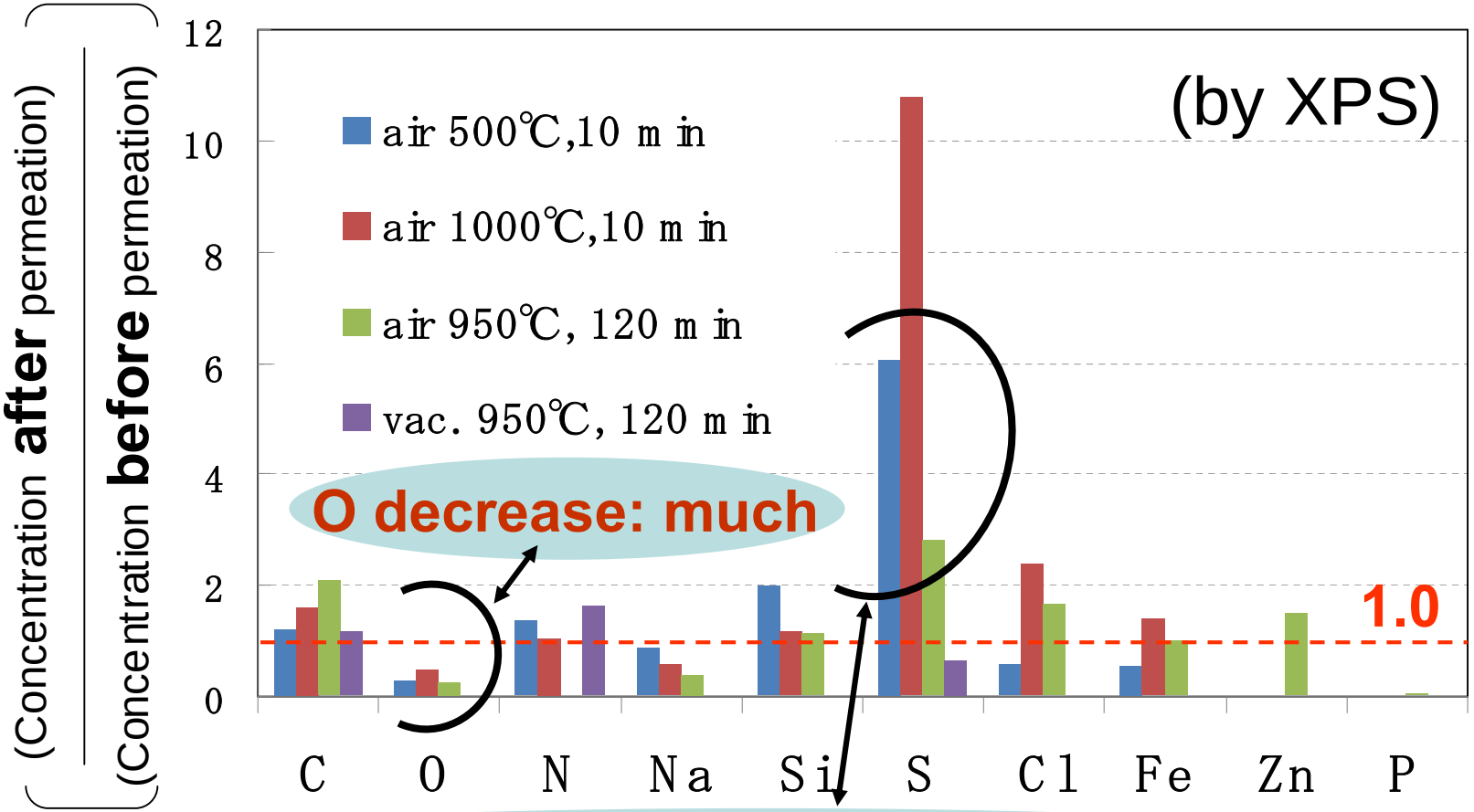
Initial Permeation rate & surface S concentration



S blocks dissociative adsorption of D2

Change of surface elemental concentration *due to D_2 permeation*



Increase of S is remarkable for the air- annealed samples

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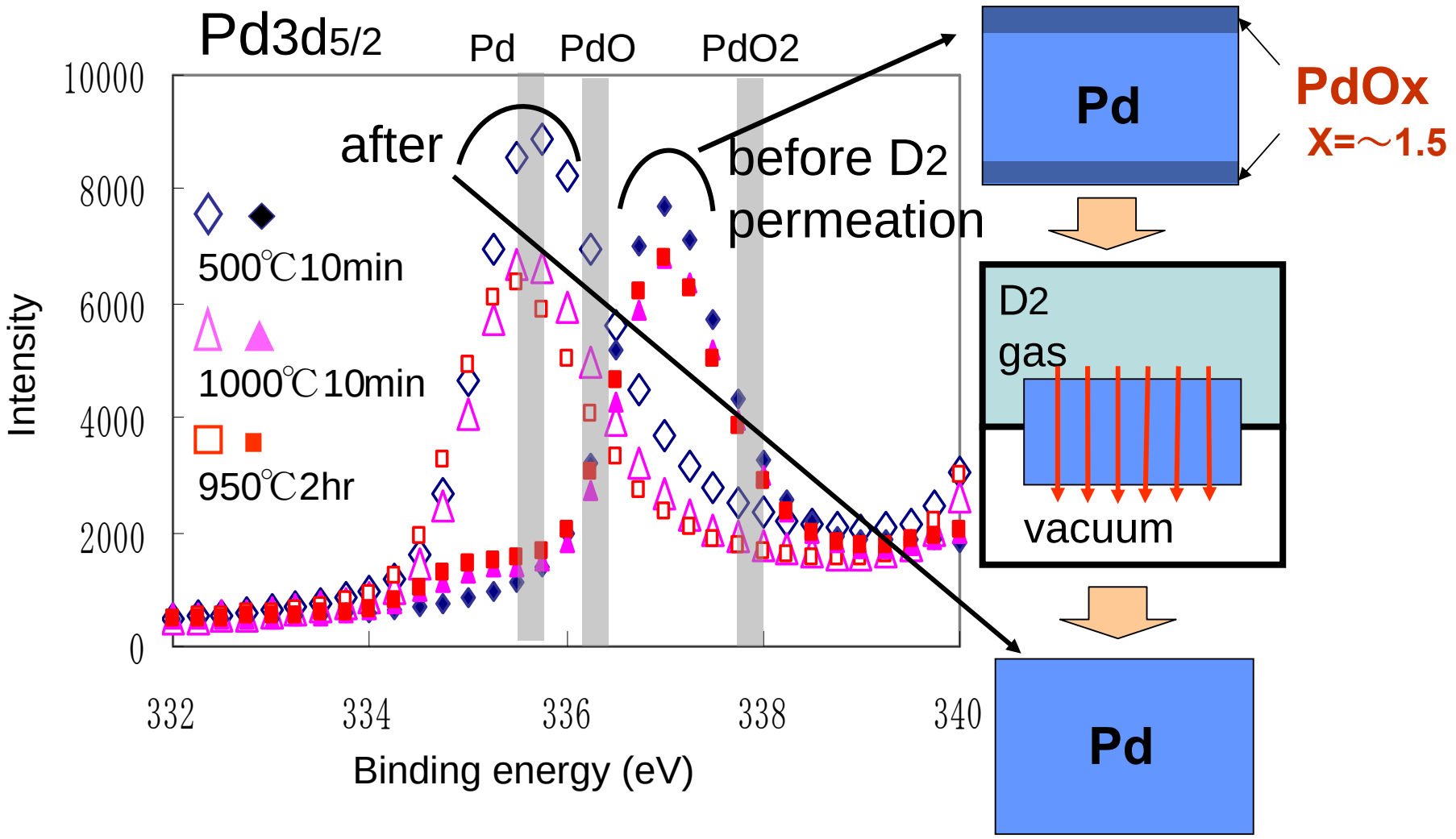
Reduction of PdOx by D2 Permeation



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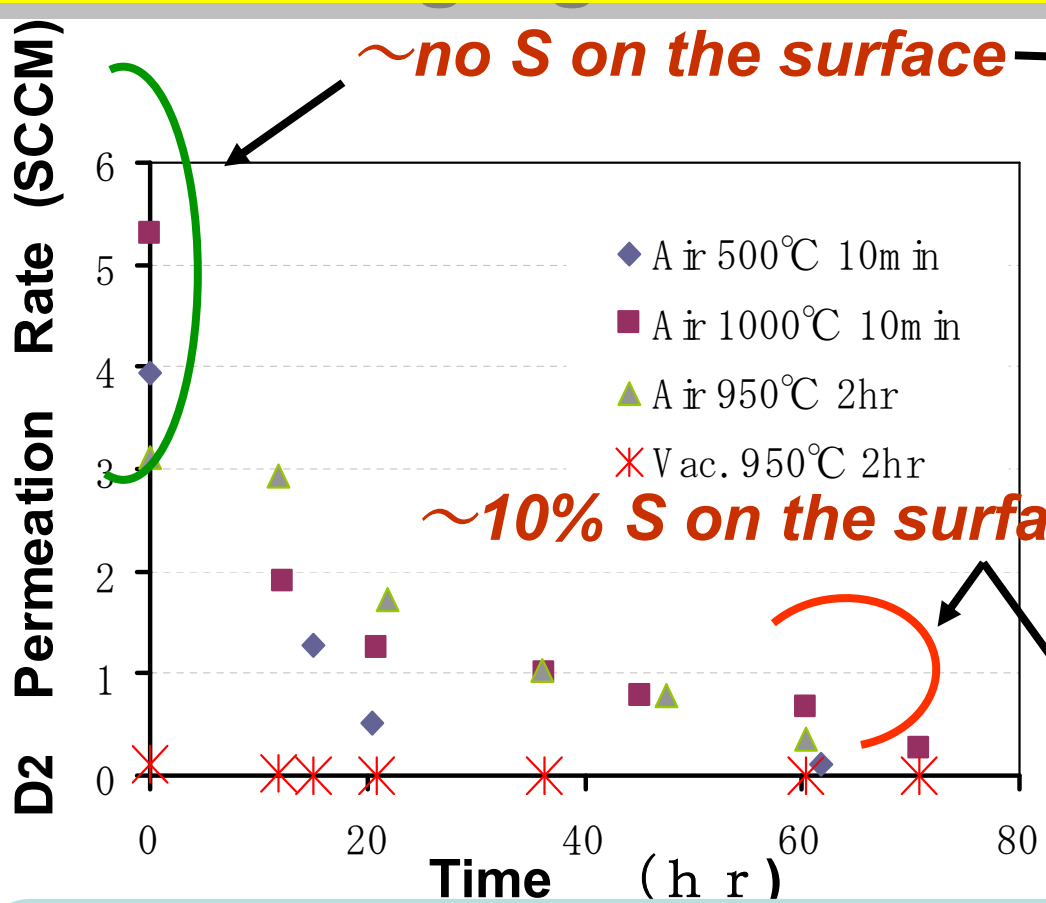
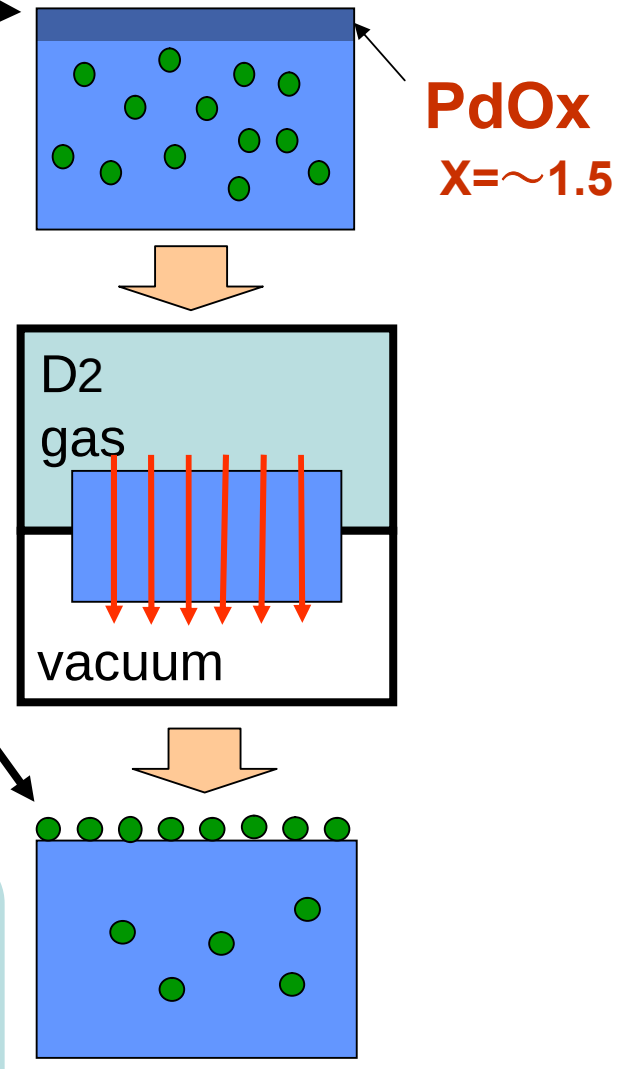
Surface segregation of S by D2 permeation





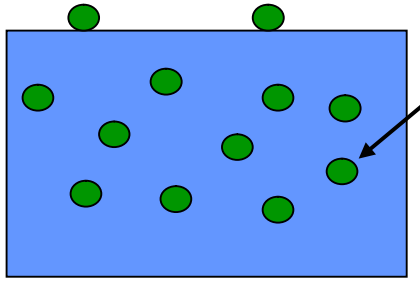
With D2 permeation:

1) PdOx is reduced to Pd

2) S segregates to the surface @70℃

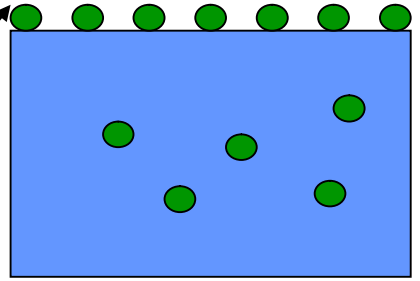
Pre-treatment of Pd for improving D_2 permeation rate

As-purchased Pd

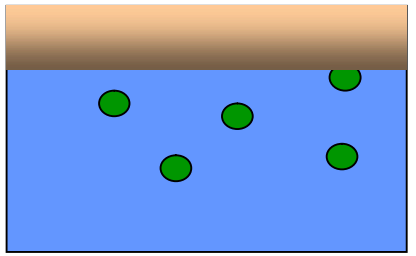


Annealing in vacuum at high temperature

- S segregates to the surface



Annealing in air at high temperature



- surface- segregated S is removed
- Pd surface layer is oxidized

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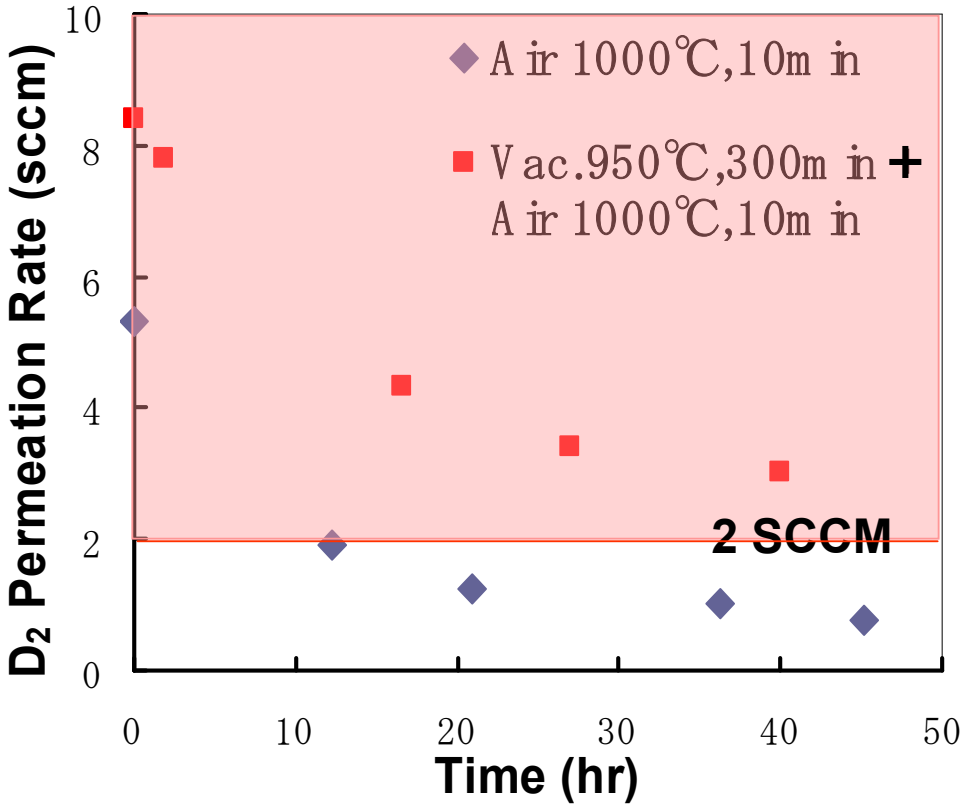
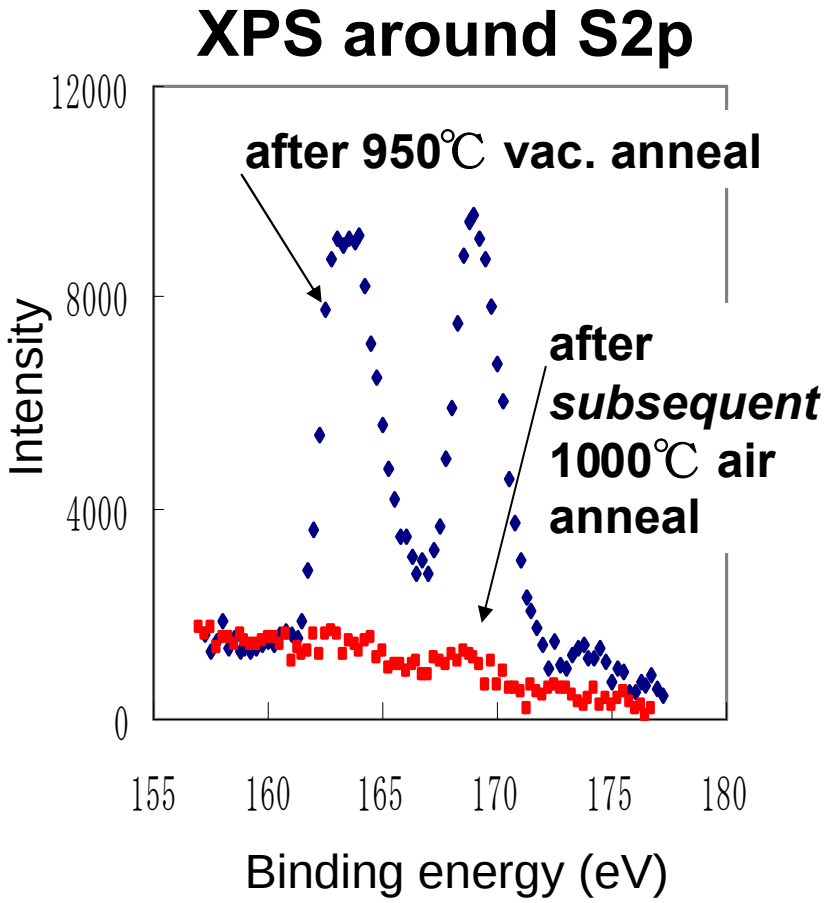
D2 permeation rate vs time for Pd heat-treated in vacuum & in air





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Summary (1)

Vacuum annealing

1. By annealing in vacuum at high temperatures, S is segregated to the surface

2. Due to the segregated S, D2 permeation rate is greately decreased (S poisoning of Pd)

Air annealing

3. Annealing in air at high temperatures oxydizes Pd but removes S from the surface

Summary (2)

D2 permeation

4. With D2 permeation, oxydized Pd is reduced to metallic Pd at a temperature as low as 343K (70℃)

5. With D2 permeation, S impurity in Pd bulk is segregated to the surface at a temperature as low as 343K (70℃)

Improving D2 permeation rate

6. By combining vacuum- and air-annealing, a high permeation rate is obtained and its degradation is comparatively small.