

Determination of hydrogen concentration in Zircaloy cladding using hot vacuum extraction method with two-step heating



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- The amount of hydrogen absorbed to the fuel cladding increases by extended burnup fuel.
- The hydrogen absorbed to the metal phase of fuel cladding which strongly influences the cladding embrittlement.
- It is important to measure the hydrogen content only in the metal phase to estimate the embrittlement of cladding.

Separately measurement was applied for the hydrogen content in the metal phase and oxide layer



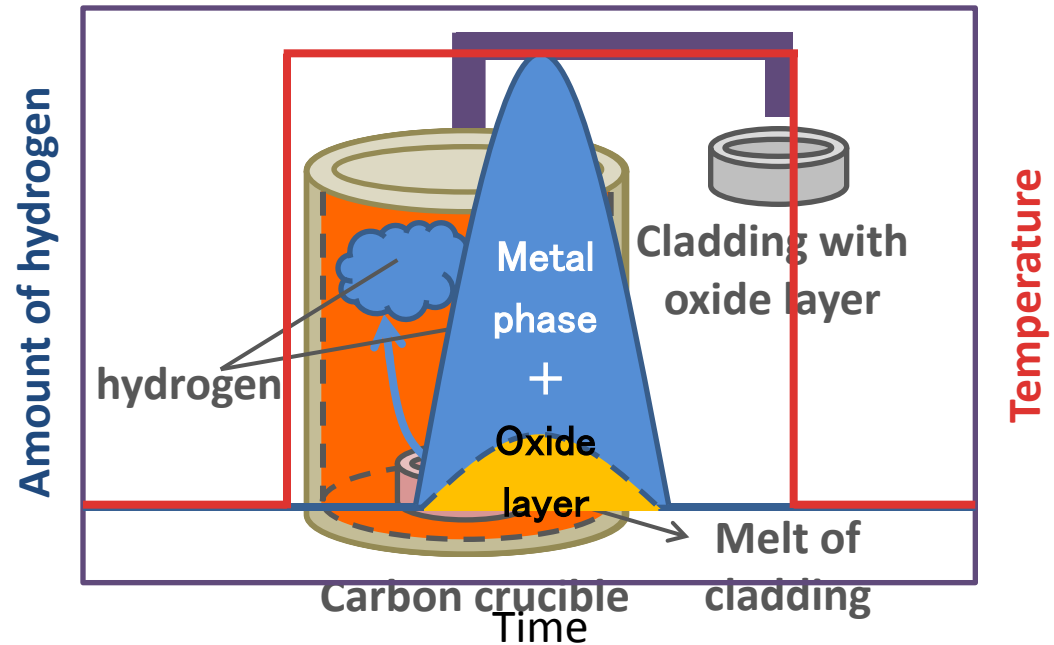
two-step heating method
(developed by PSI)¹⁾

¹⁾ A. Hermann, et al., "Hydrogen distribution between fuel cladding metal and overlying corrosion layers," Proc. Int. Topical Meeting on LWR fuel performance, Park City, USA, 372–384 (2000).

What is the two-step heating method?

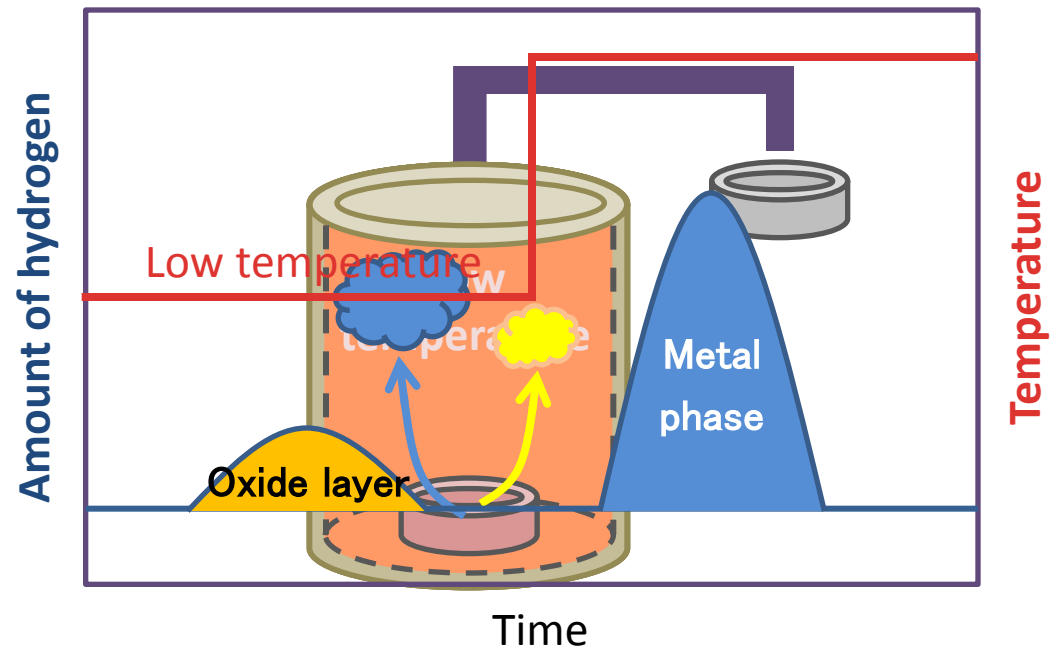
- Previous method (one step)

It is impossible to measure the amount of hydrogen only in the metal phase



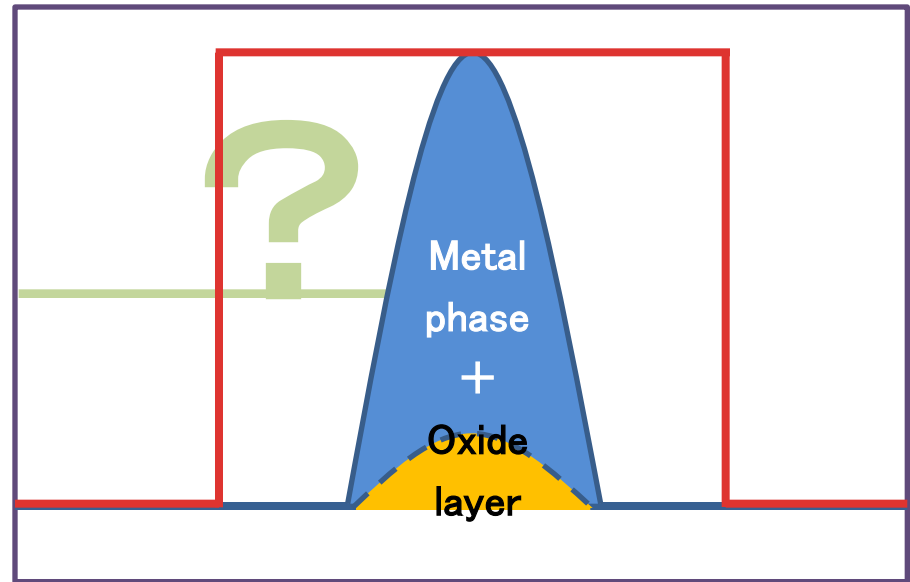
- Two-step heating method

It is possible to measure hydrogen in the metal phase and oxide layer separately



The temperature of first step is strongly related for the property of the oxide layer

Amount of hydrogen



Temperature

The two-step heating method is modified



Determination procedure of the heating temperature to release the hydrogen in the oxide layer (Lower side temperature)

Temperature

Experimental procedure

① Obtain hydrogen release curve by continuous heating



② Optimization of hydrogen release temperature from oxide layer



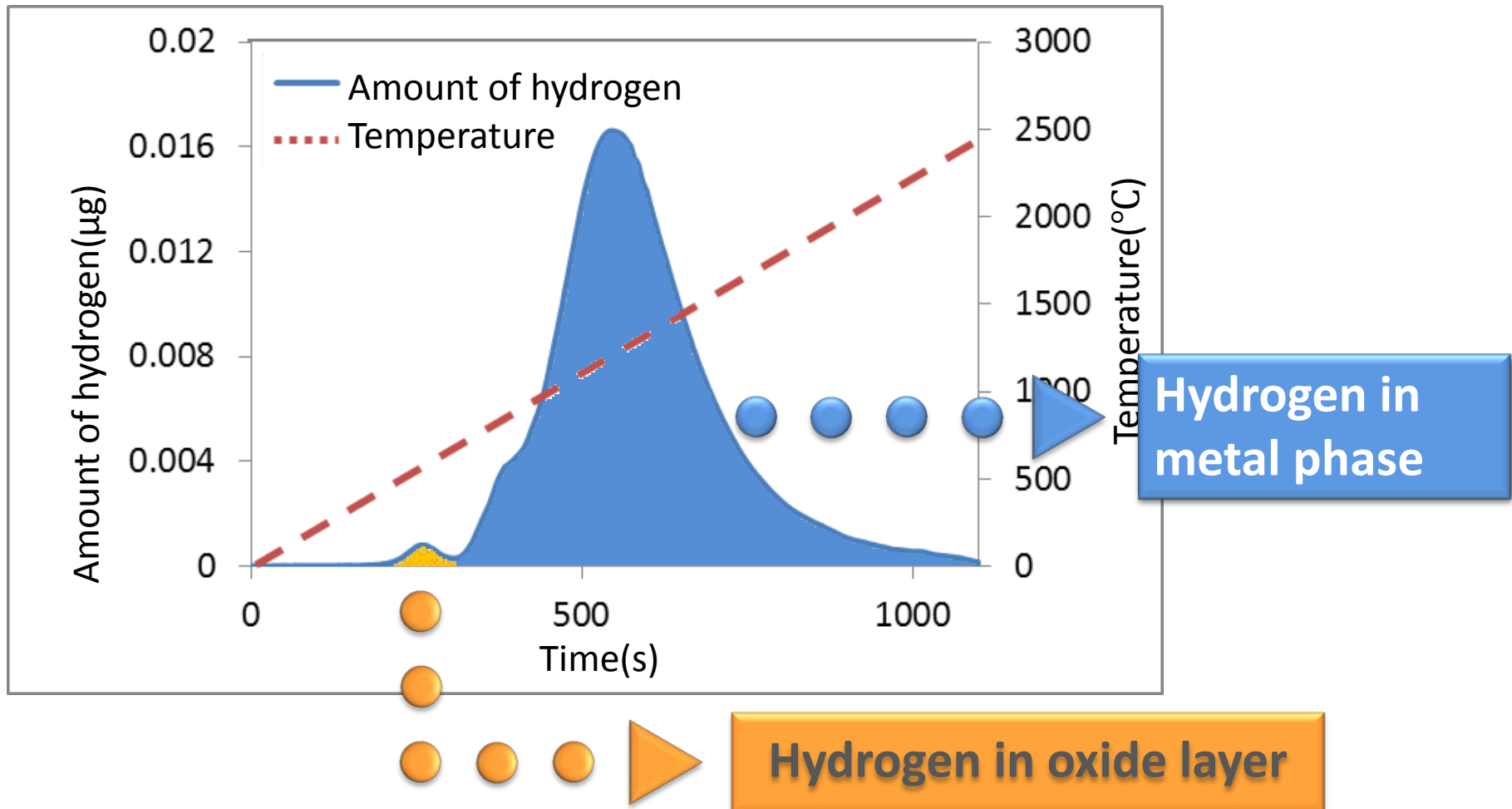
③ Two-step heating (unirradiated cladding)



④ Adaptation to irradiated cladding

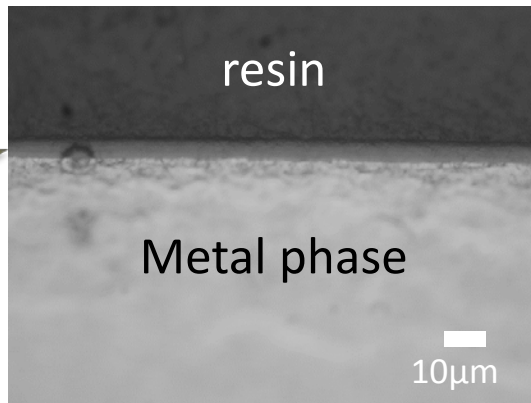
① Obtain hydrogen release curve by continuous heating

Continuous heating was performed to guess temperature of hydrogen release in the oxide layer. Measurement time is 1100 seconds and measurement temperature is rising to 2500°C from room temperature.

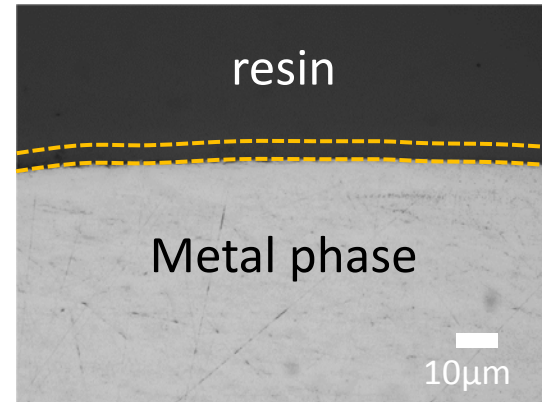


Confirmation of hydrogen release behavior from oxide layer

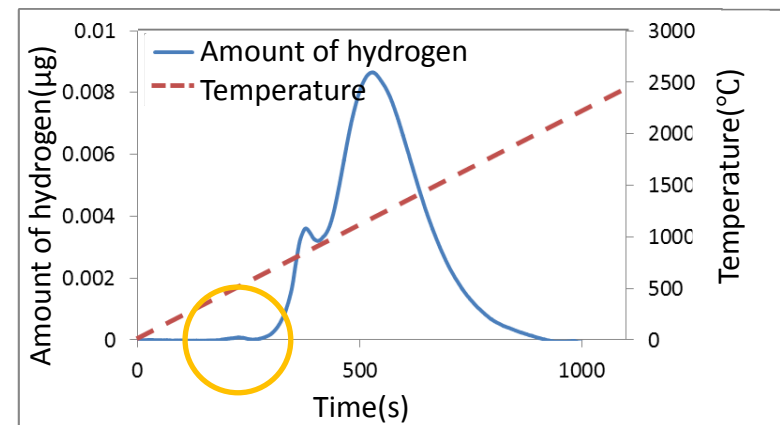
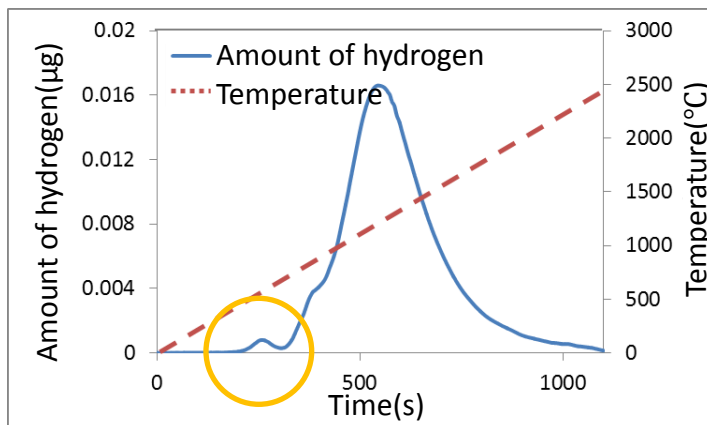
Oxide layer



Cladding with oxide layer



Without the oxide layer



Experimental procedure

① Obtain hydrogen release curve by continuous heating



② Optimization of hydrogen release temperature from oxide layer

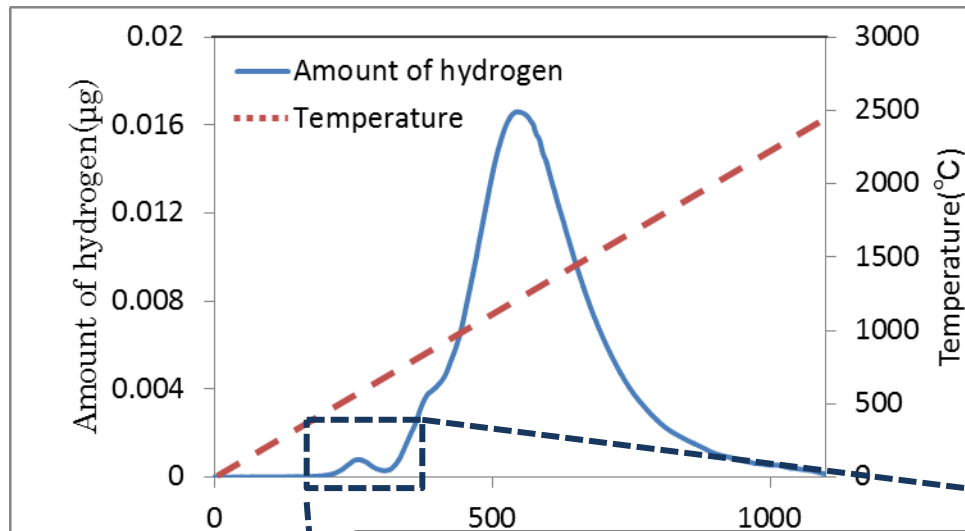


③ Two-step heating (unirradiated cladding)



④ Adaptation to irradiated cladding

② Optimization of hydrogen extraction temperature from oxide layer

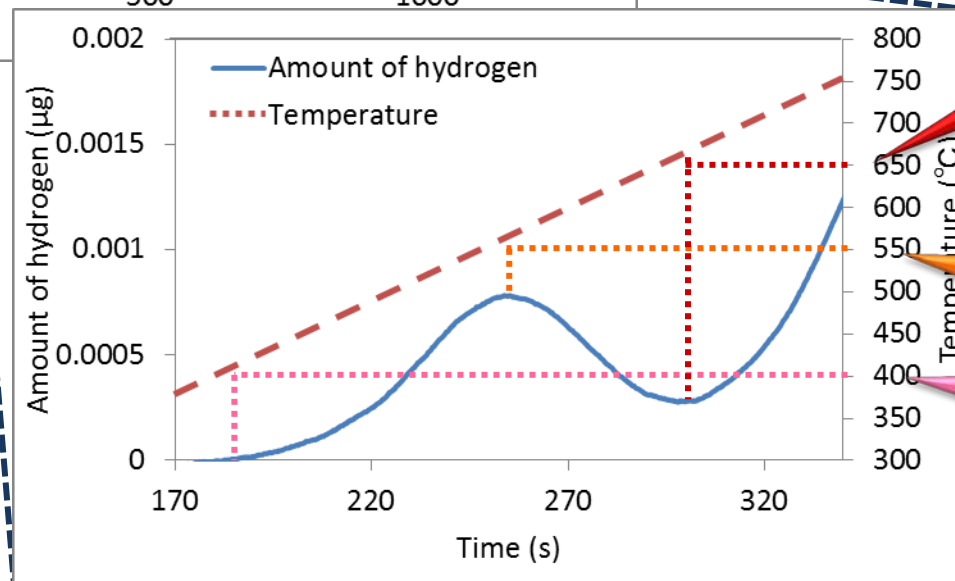


It's determined the optimum temperature to measure the hydrogen separately

A : beginning of the peak (400°C)

B : Top of the peak (550°C)

C : End of the peak (650°C)



C : 650°C

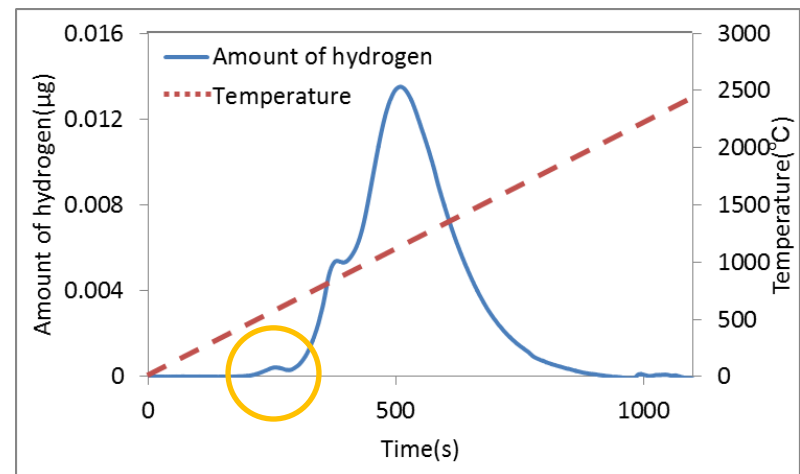
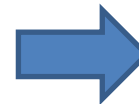
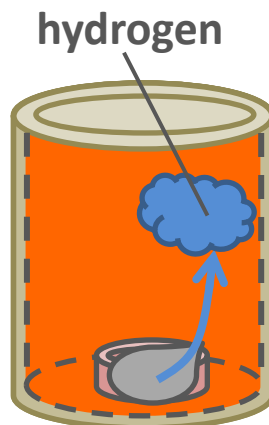
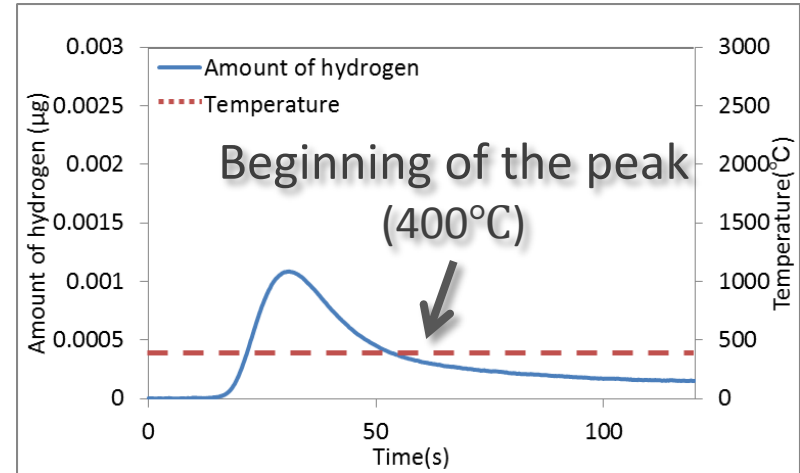
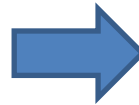
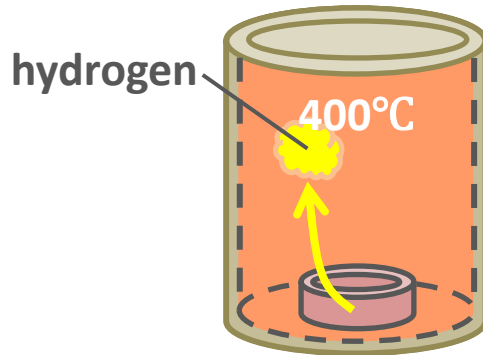
B : 550°C

A : 400°C

Determination of the temperature for separation measurement

It's confirmed a peak at low temperature side in the result of continuous heating.
 → **The hydrogen in the oxide layer was not fully release at 400°C.** (A,B,C).
 It's decided optimum temperature of first step in two-step heating method.

A:400°C
 B:550°C
 C:650°C

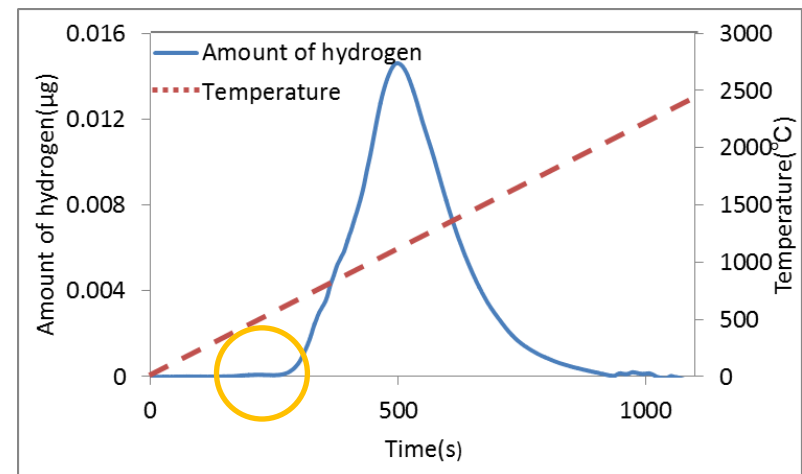
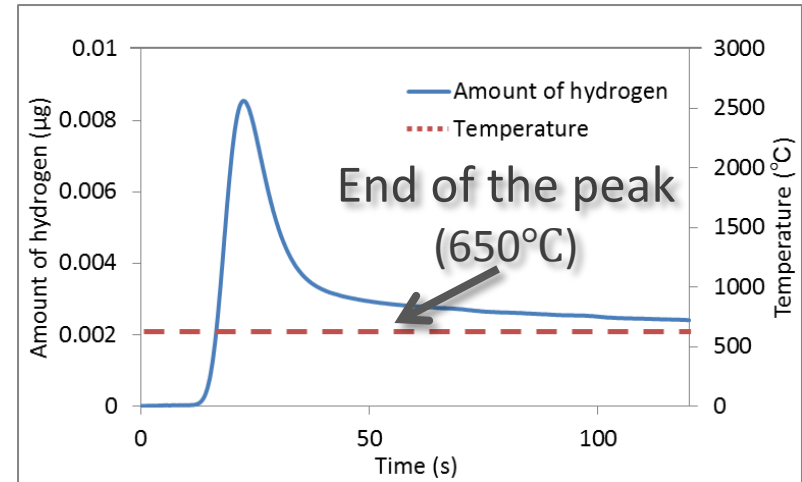
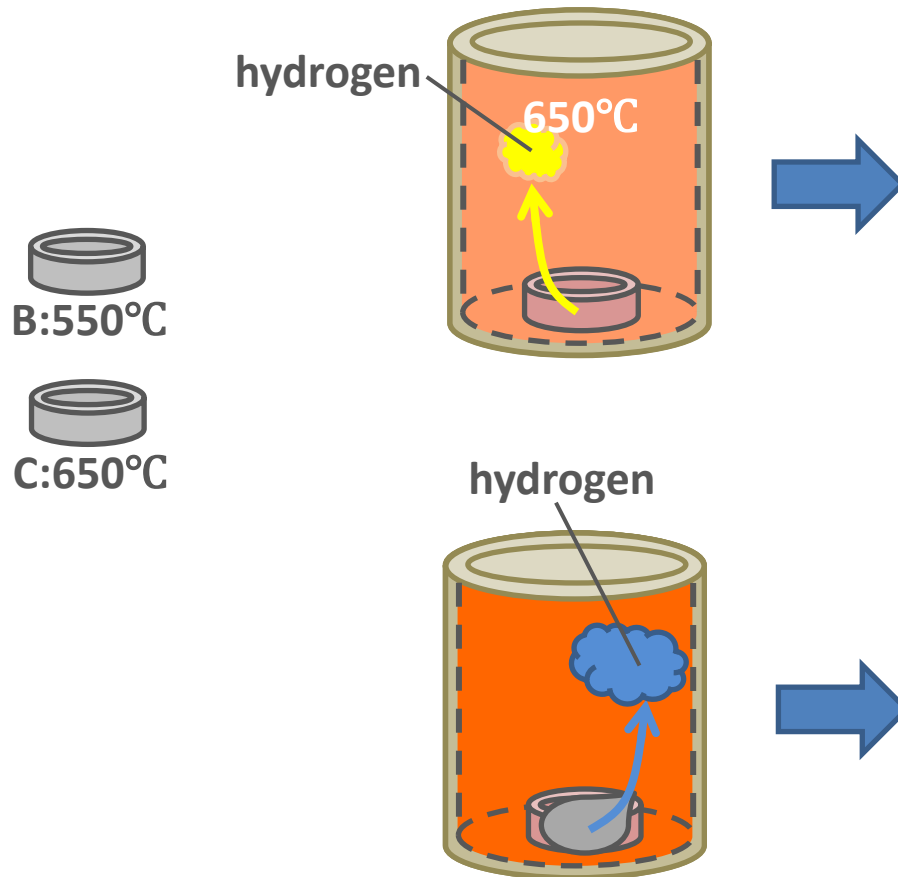


Determination of the temperature for separation measurement

There's no peak at low temperature side in the result of continuous heating.

→ The hydrogen in the oxide layer was fully release at 650°C.

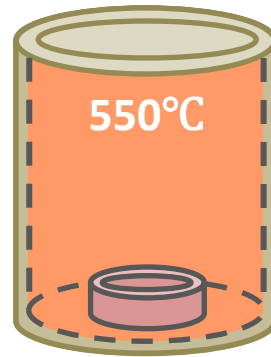
But, the hydrogen in metal phase was released at 650°C



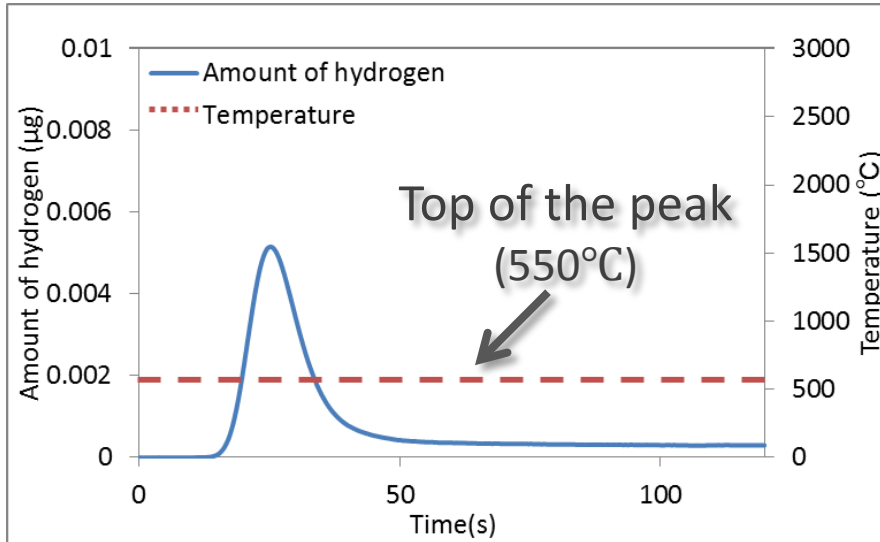
Determination of the temperature for separation measurement



B:550°C

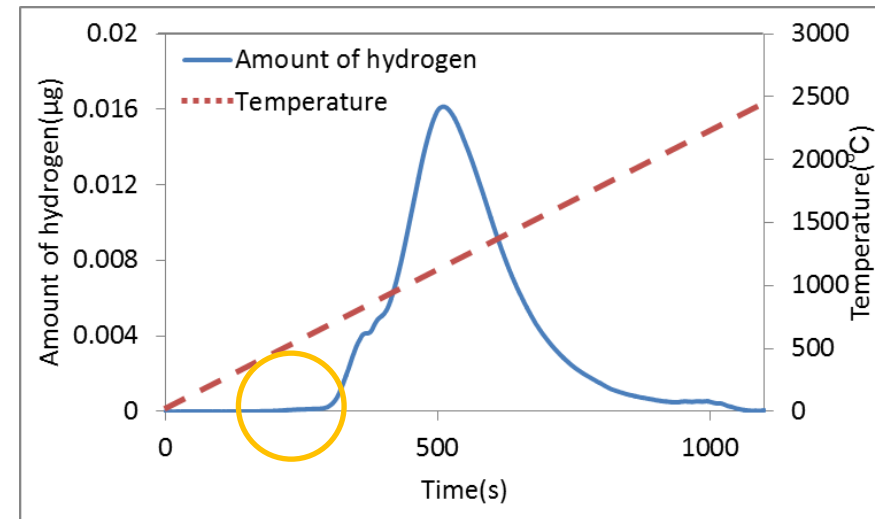


B : In the case 550°C (Top of peak)



The hydrogen only in the oxide layer was fully released at 550°C.

There's no peak at low temperature side.



Temperature of **peak top** is configured the temperature of first step, It was guessed to be possible to measure hydrogen separately.

Experimental procedure

① Obtain hydrogen release curve by continuous heating



② Optimization of hydrogen release temperature from oxide layer



③ Two-step heating (unirradiated cladding)

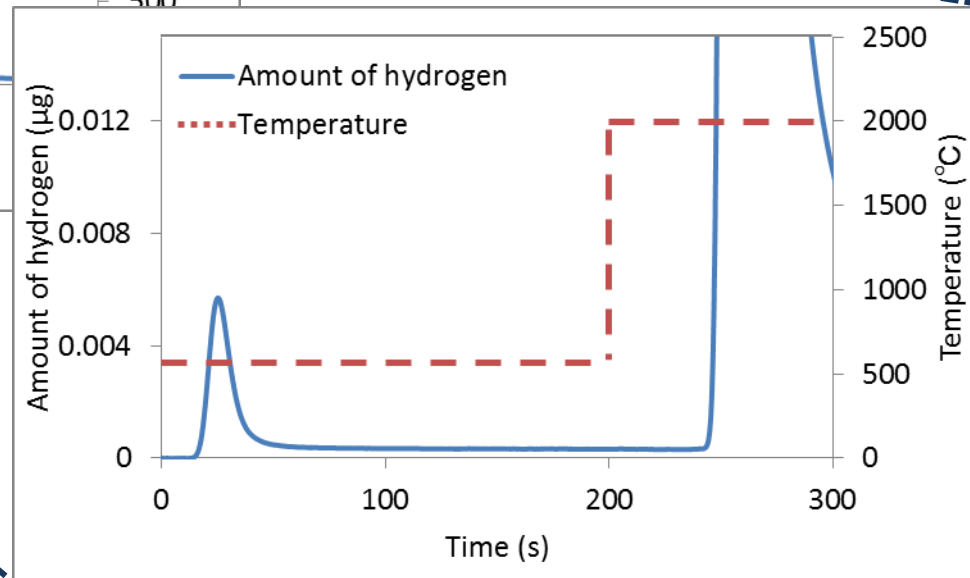
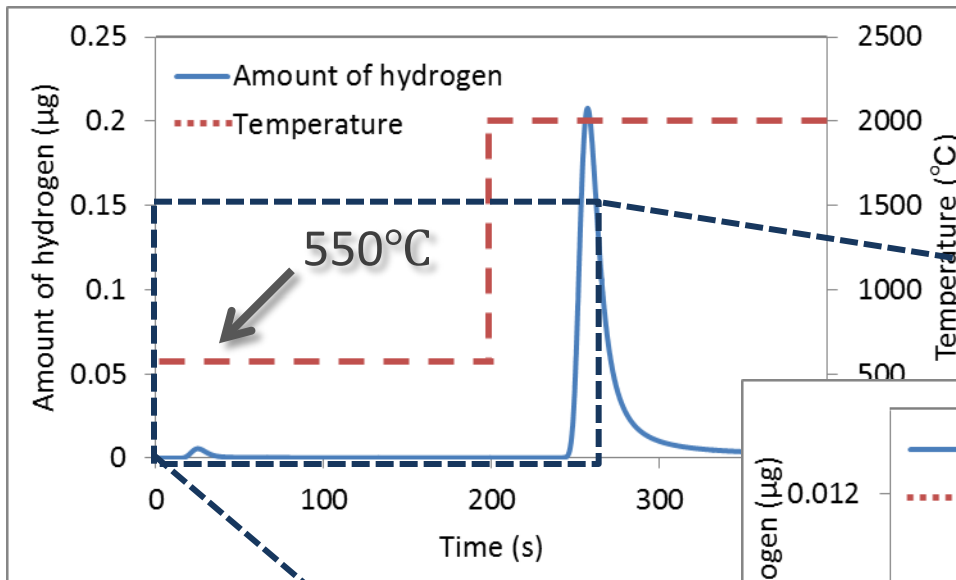


④ Adaptation to irradiated cladding

③ Two-step heating (unirradiated cladding)

It's confirmed the hydrogen in oxide layer was released at the first step and the hydrogen in metal phase was released at the second step.

It's possible to measure the hydrogen separately.



Experimental procedure

① Obtain hydrogen release curve by continuous heating



② Optimization of hydrogen release temperature from oxide layer

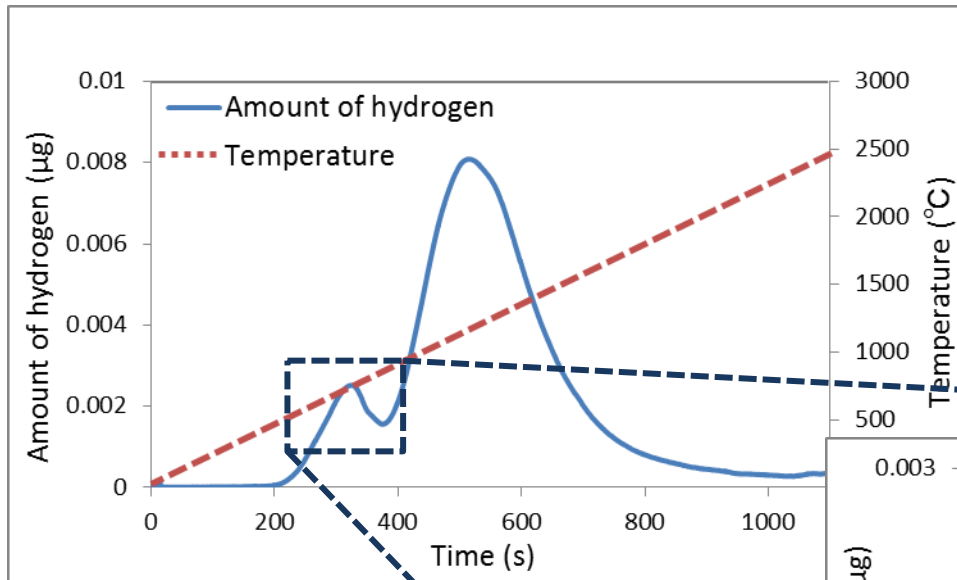


③ Two-step heating (unirradiated cladding)



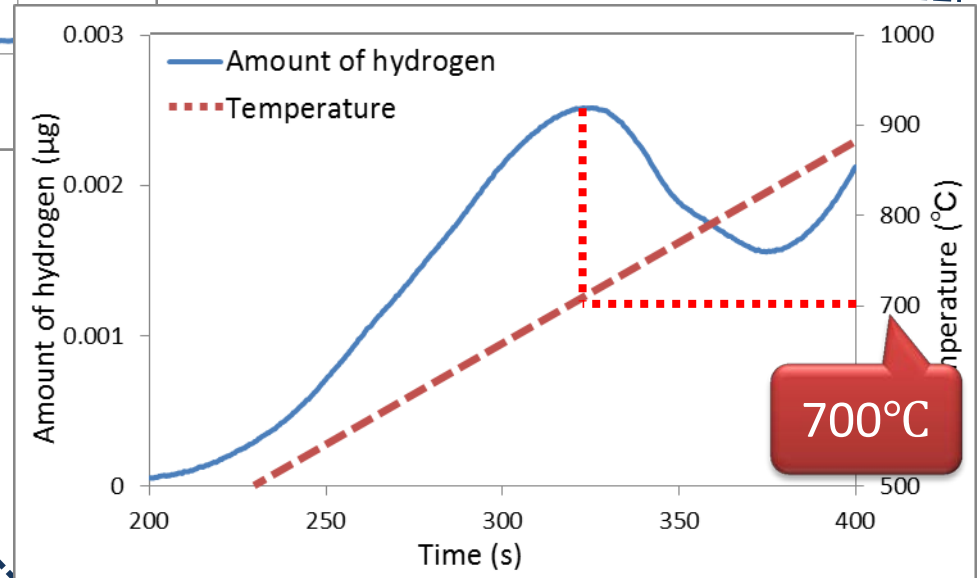
④ Adaptation to irradiated cladding

④Adaptation for irradiated cladding



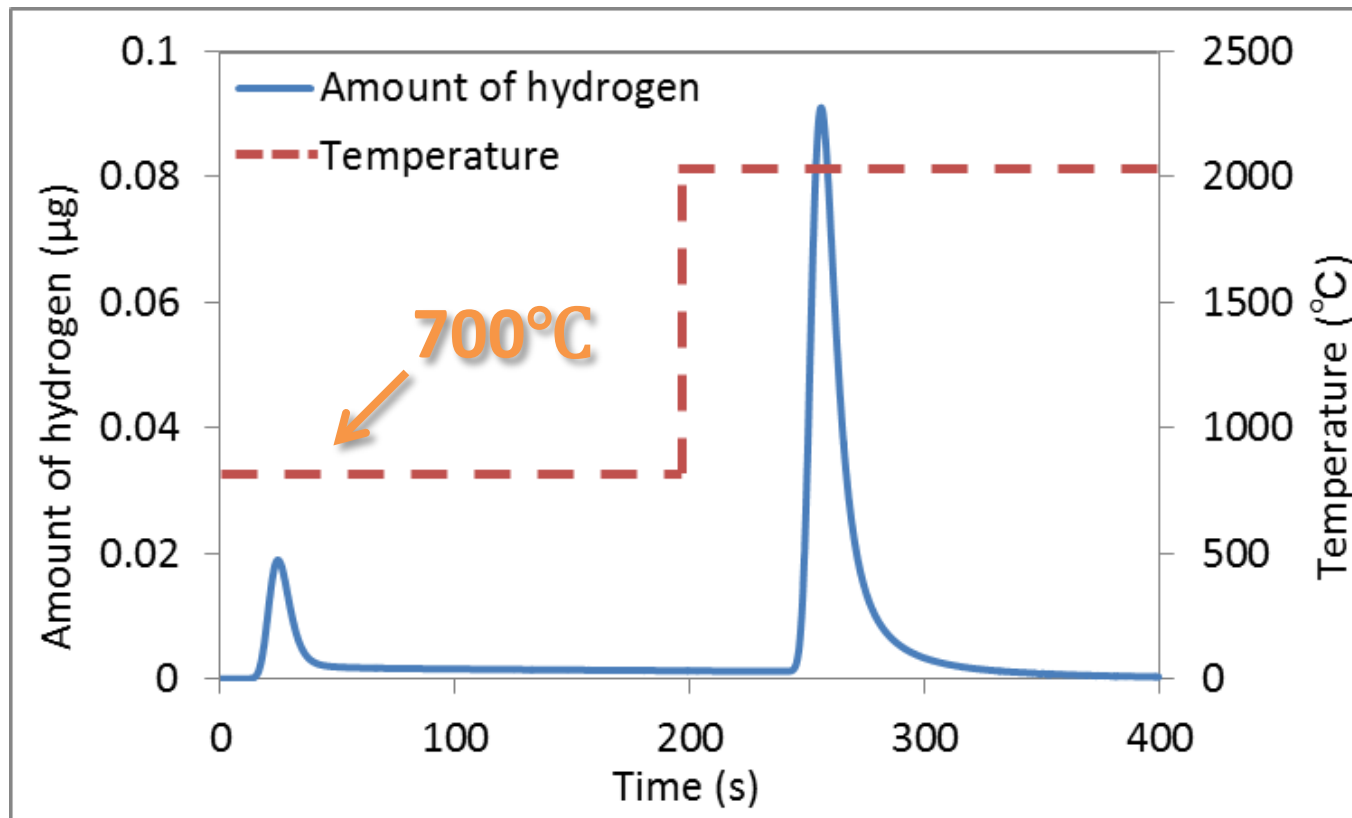
①Obtain hydrogen release curve by continuous heating

②Optimization of hydrogen release temperature from oxide layer



④Adaptation for irradiated cladding

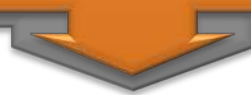
③Two-step heating(Irradiated cladding)



The hydrogen in the irradiated claddings can be measured separately.

Conclusion

- Determination procedure of the heating conditions on the two-step heating method was modified.
- The results of confirmation test, this procedure is valid for irradiated cladding.



This method is useful for the estimation for embrittlement of irradiated cladding.