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Quantitative Evaluation of Hydrogen Embrittlement of Metal Membrane Detected by *In-Situ* Small Punch Test under Hydrogen Permeation

Prof. Yoshihisa Matsumoto, Dr. Eng.,
Oita National College of Technology, Japan,

Prof. Hiroshi Yukawa, Dr. Eng.,
Nagoya University, Japan,

Prof. Tomonori Nambu, Dr. Eng.,
Suzuka National College of Technology, Japan

Background

Recently,
Pd-Ag alloys were used
for hydrogen purification

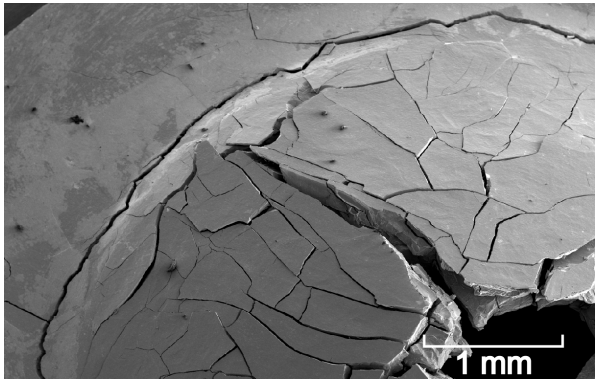
Disadvantages:
× Low hydrogen permeability
× Low strength at high temp .
× High cost (noble metal)

non-Pd system

Niobium (Nb), Vanadium (V) and their alloys:

- ○ Higher/Highest hydrogen permeability
- ○ High temperature strength
- ○ Bountiful resources
- ○ Low material cost ($55/kg for Nb)

  However, poor resistance to hydrogen embrittlement



Brittle fracture morphology
of hydrogen dissolved Nb.

For practical use;

The high hydrogen permeability, strong resistance to hydrogen embrittlement and durability should be encouraged.

Objective

The aim of the performed work is;
(1) To clarify the hydrogen embrittlement of niobium, vanadium and its alloy membranes quantitatively under ⋯

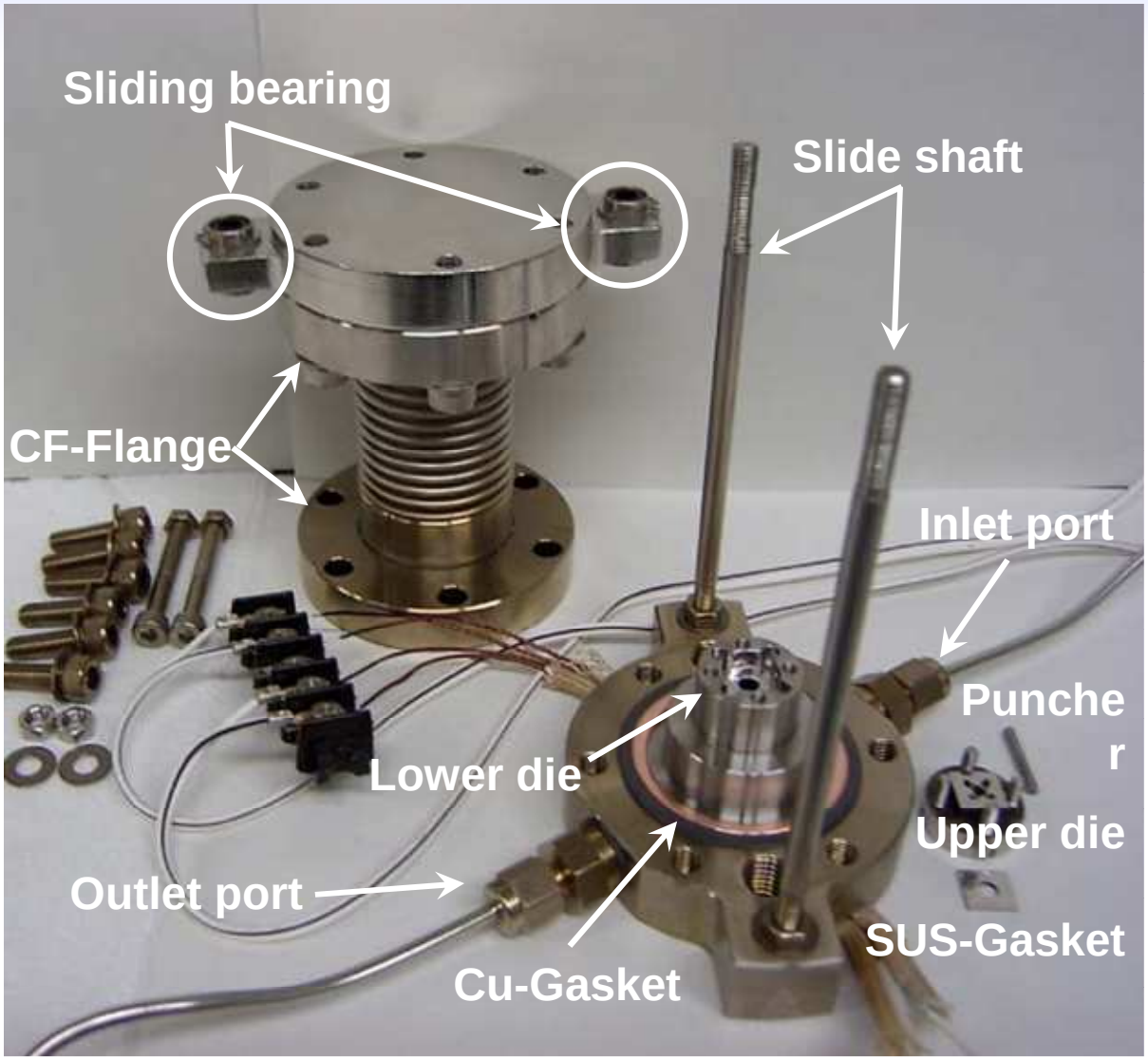
the hydrogen dissolution and permeation conditions by using an *in-situ* Small Punch (SP) test apparatus.

(2) The ductile-to-brittle transition hydrogen concentrations (DBTC) of them are also evaluated from SP absorption energies of the samples tested under ⋯

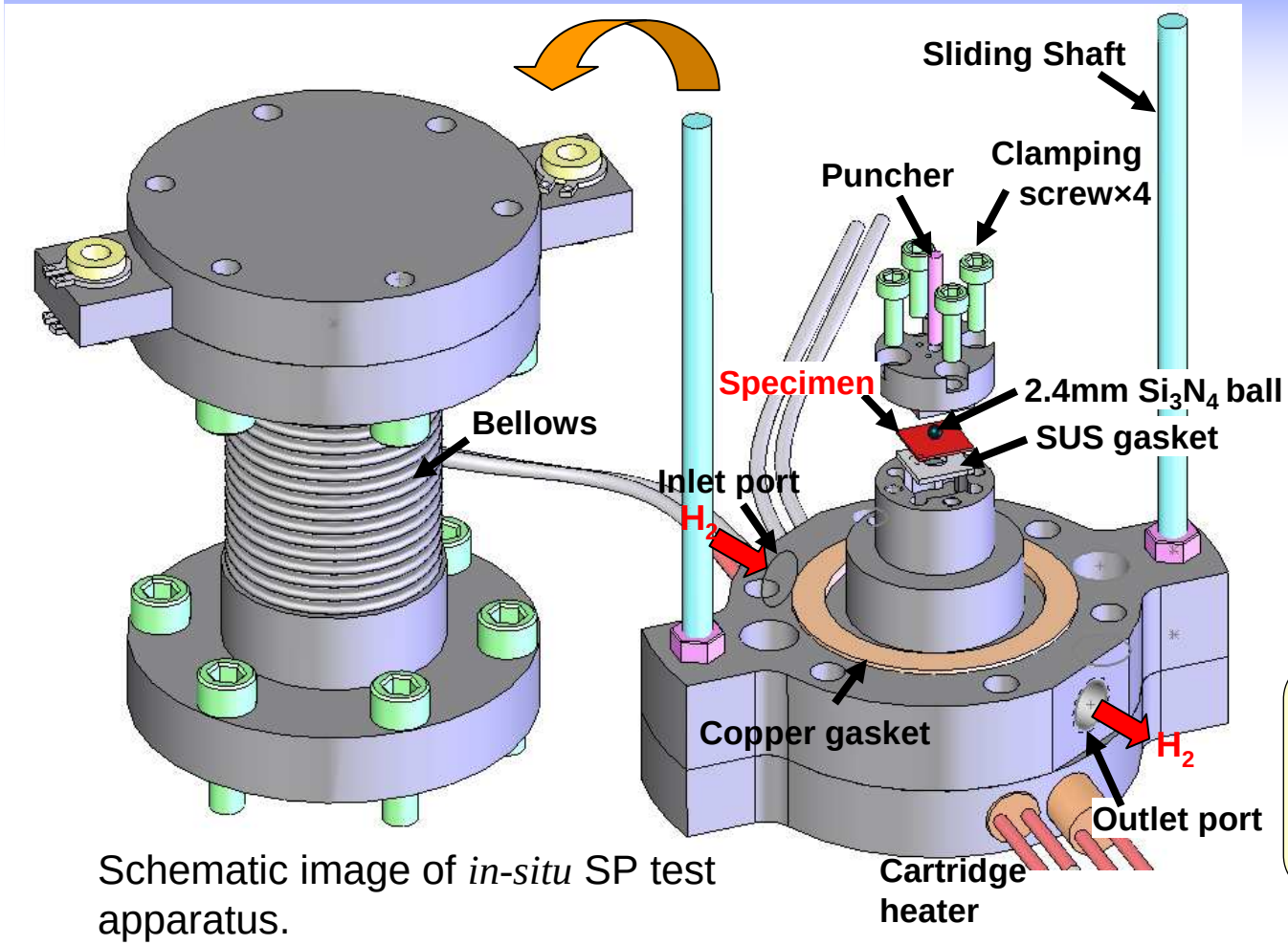
various hydrogen dissolution conditions.

Development of a new *in-situ* small punch test apparatus

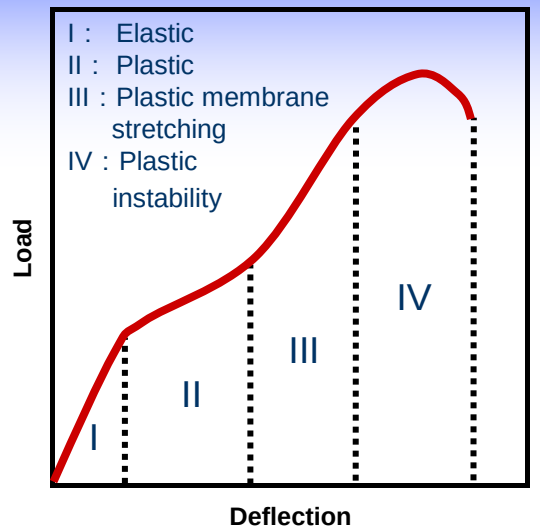
Appearance of developed *in-situ* SP test apparatus equipped with a gas flow system.



In-situ SP test apparatus with gas flow system



Schematic image of *in-situ* SP test apparatus.

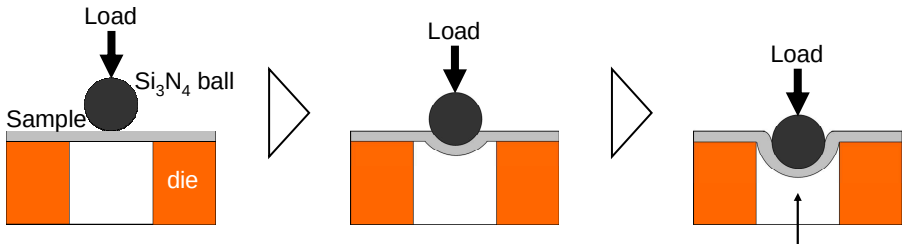


Typical load vs. deflection curve of ductile material.

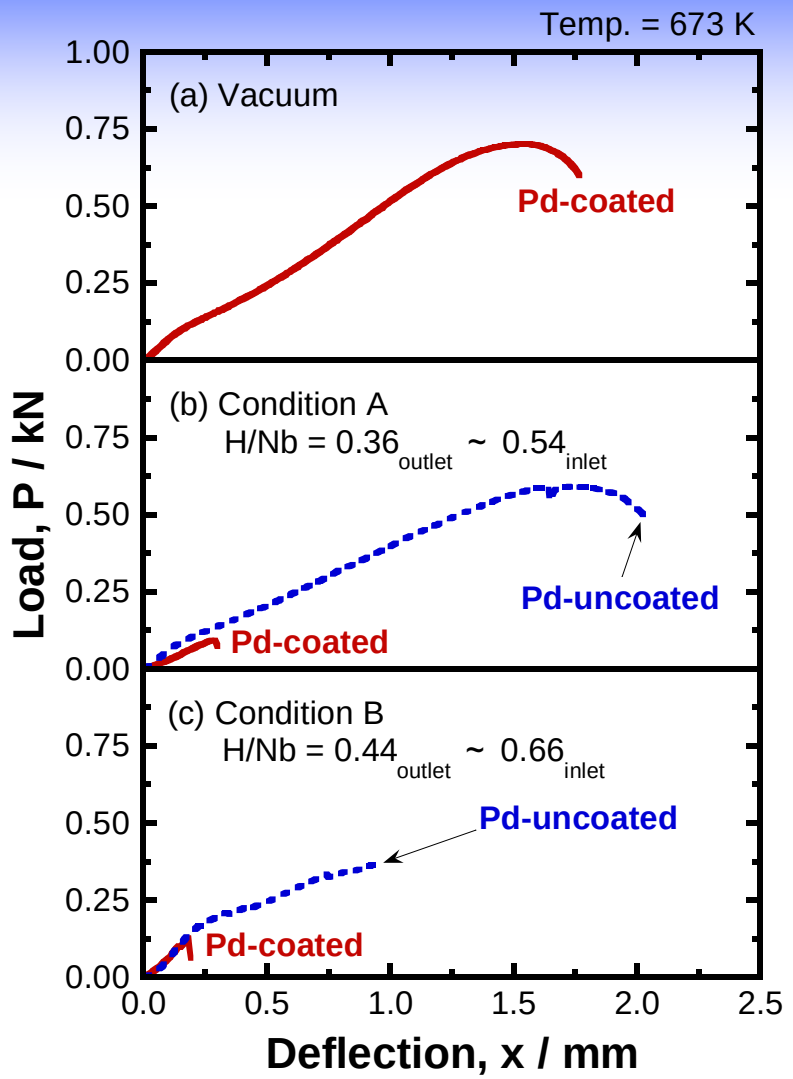
A load vs. deflection curve can be measured under either the hydrogen permeation or the constant hydrogen pressure atmosphere.

Specifications

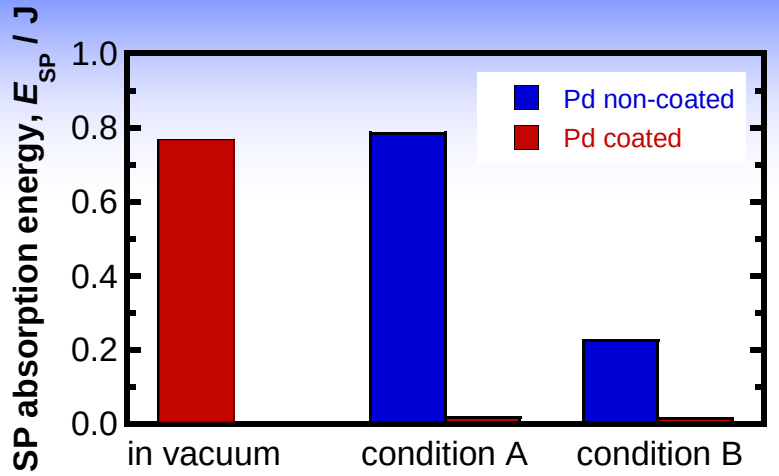
Temperature:	R.T. to 600□
Pressure:	vacuum ~ 300kPa
Atmosphere:	vacuum, H_2, etc.



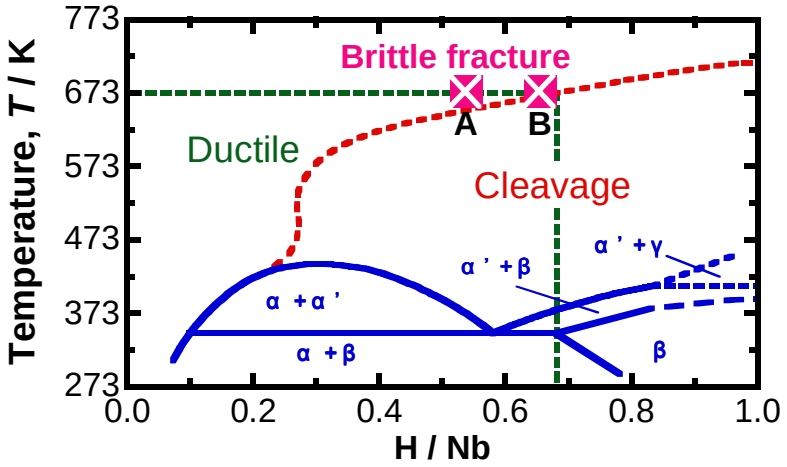
Results of *in-situ* SP test under hydrogen permeation



Load vs. deflection curves of Pd-coated and Pd-uncoated Nb specimens under various conditions.



SP absorption energies of Nb specimens under various conditions.

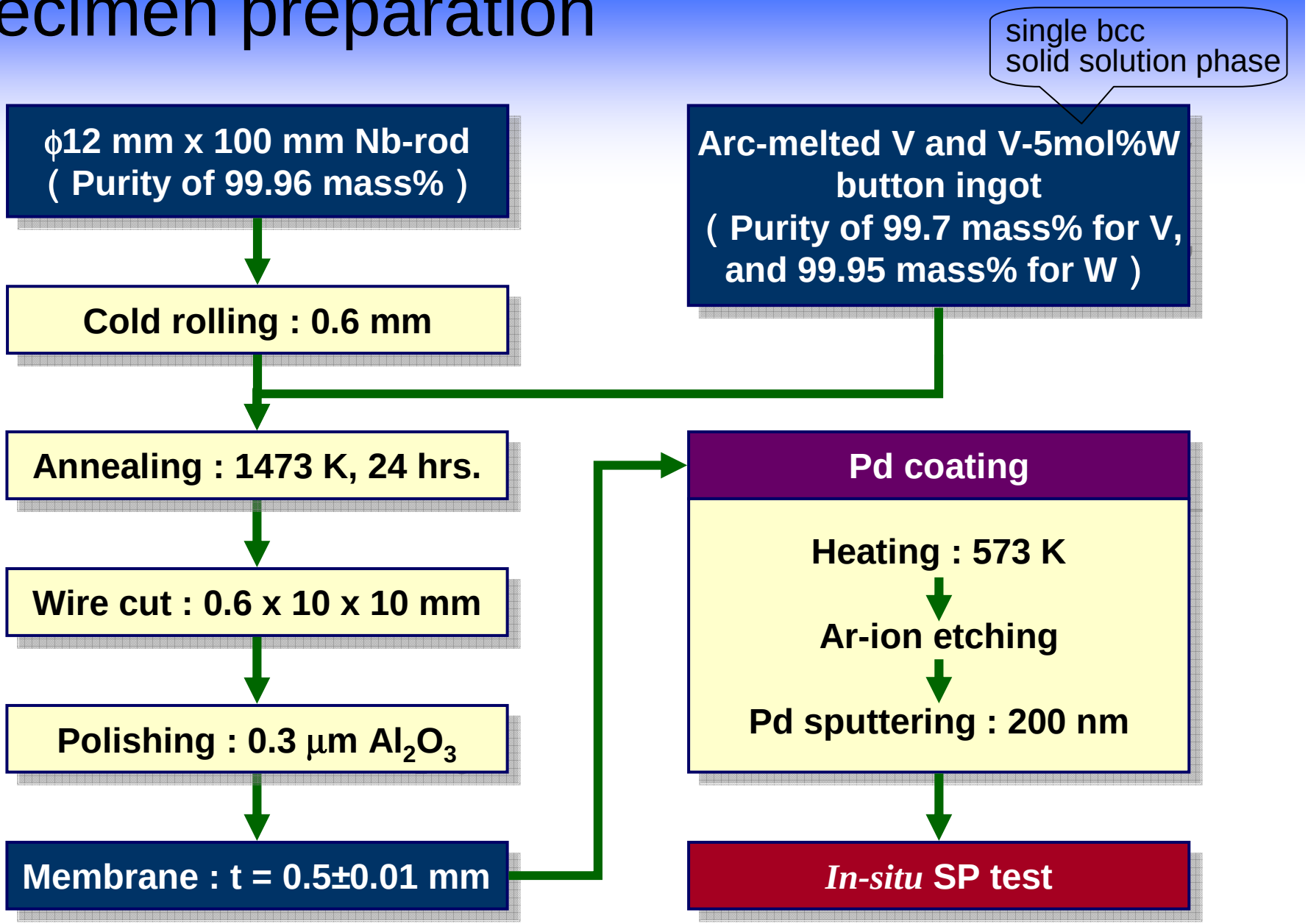


Relation between ductile-to-cleavage and hydrogen concentration.

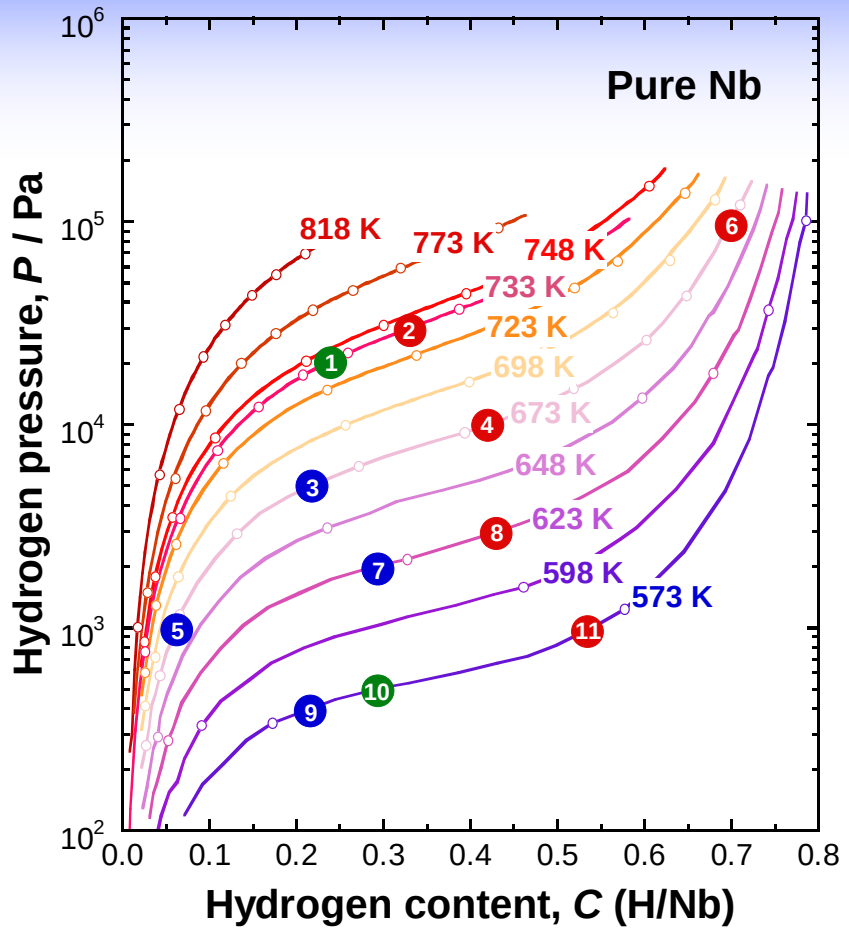
Ref. : S. Gahr and H. K. Birnbaum, Acta Metallurgica, Vol. 26 (1978) 1781.

Thus, the hydrogen sensitivity for the Pd-coated Nb metal membrane is found to be shifted greatly to lower hydrogen concentration region, as compared with Pd-uncoated Nb.

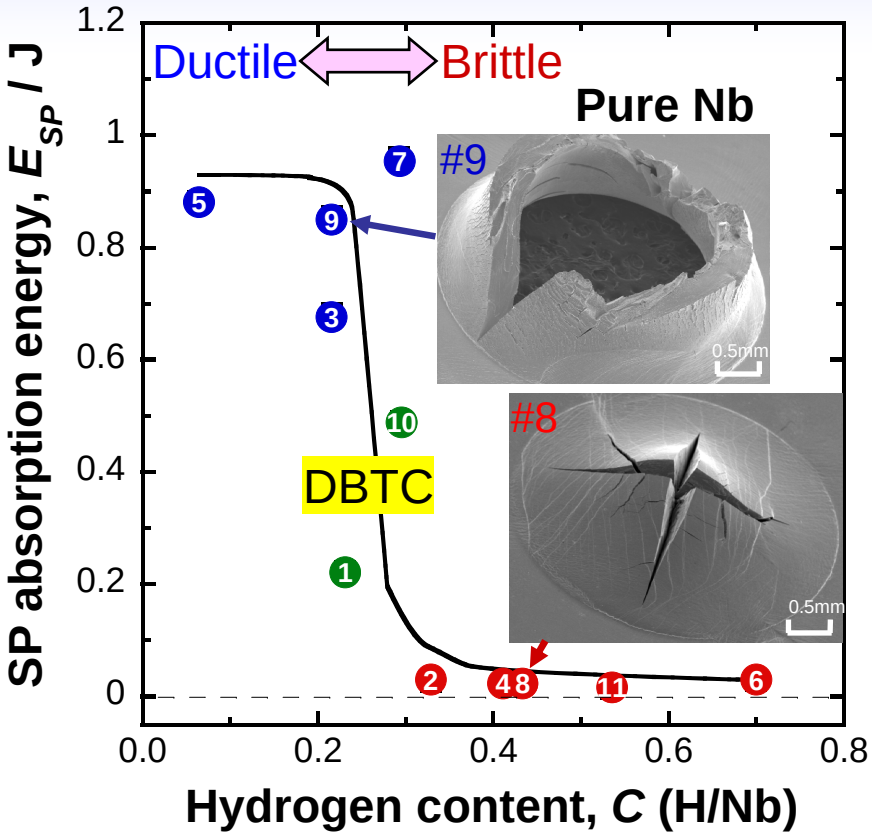
Specimen preparation



In-situ SP test conditions and absorption energies



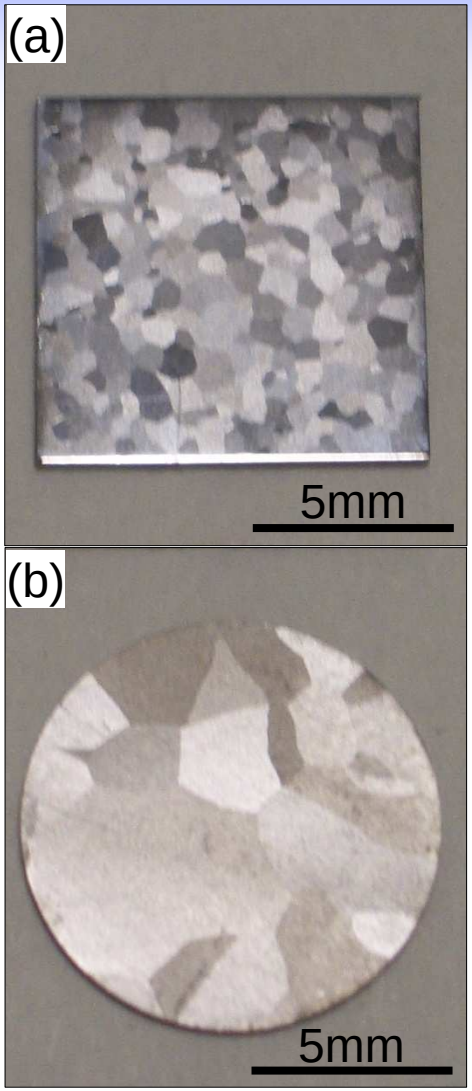
In-situ SP test conditions expressed on the PCT curves.

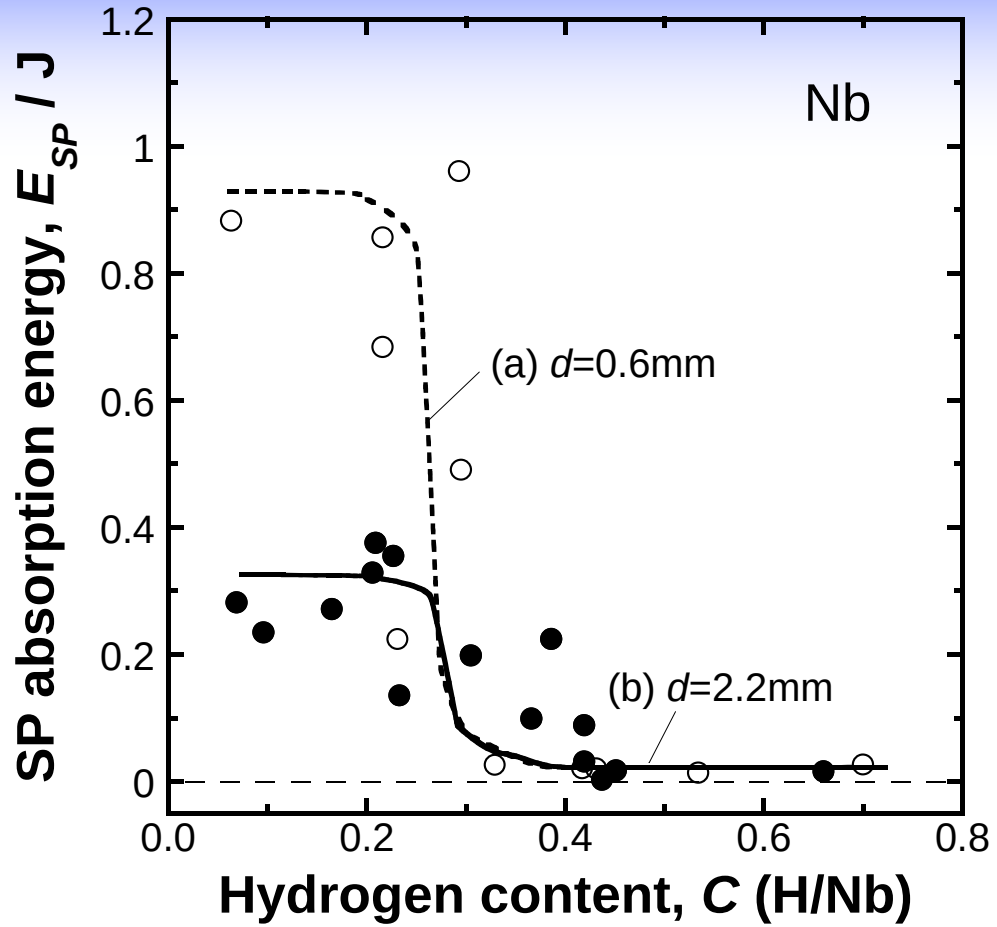


Change in the SP absorption energy with hydrogen concentration, C (H/Nb) in Pd-coated Nb matrix.

A ductile fracture occurs when the hydrogen concentration is limited less than about 0.25 in H/Nb, irrespective of temperature. Ductile-to-brittle transition hydrogen concentration (DBTC) was also discovered.

Grain size effect on DBTC



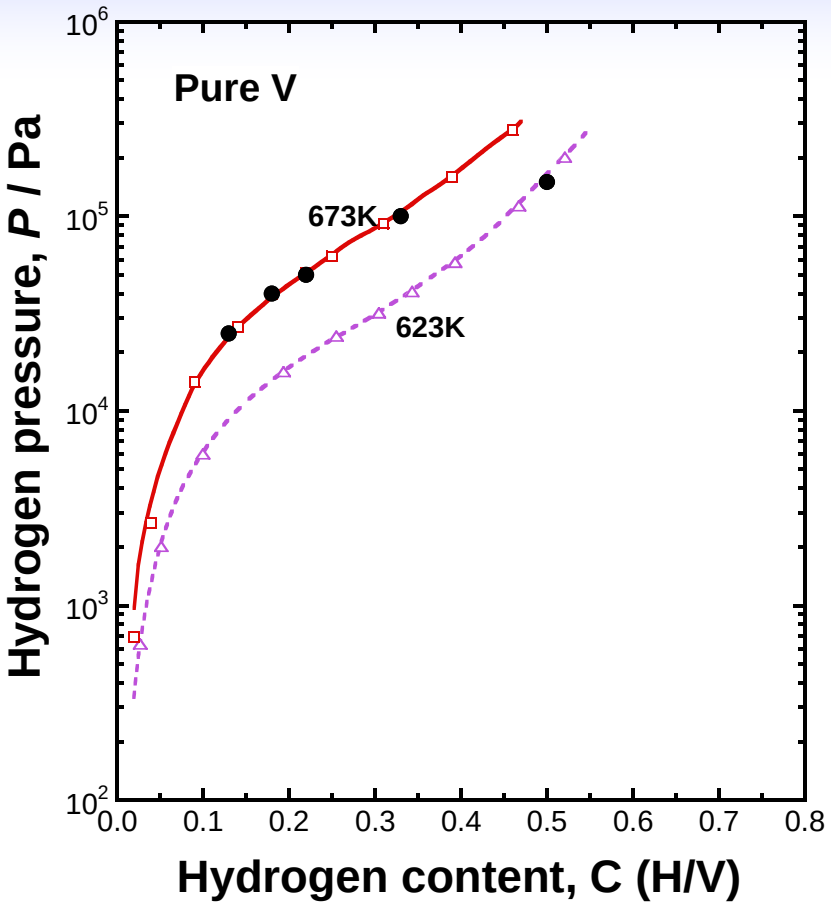


Relationship between H/Nb and SP absorption energies for different grain size, *d*, of Nb.

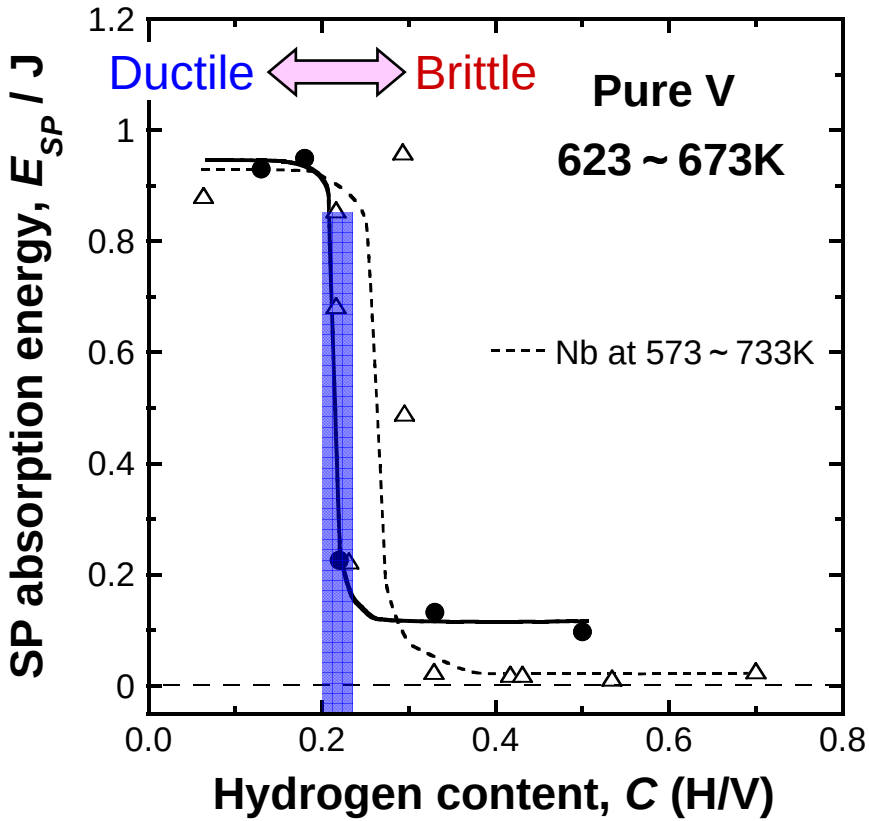
Although there is a difference in upper-shelf of SP absorption energy, it can be said that H/Nb≈0.25 is the critical concentration to brittleness.

DBTC analyses for Pd-coated pure vanadium

The similar analyses concerning the DBTC are performed to Pd-coated pure V.



In-situ SP test conditions of V expressed on the PCT curves.

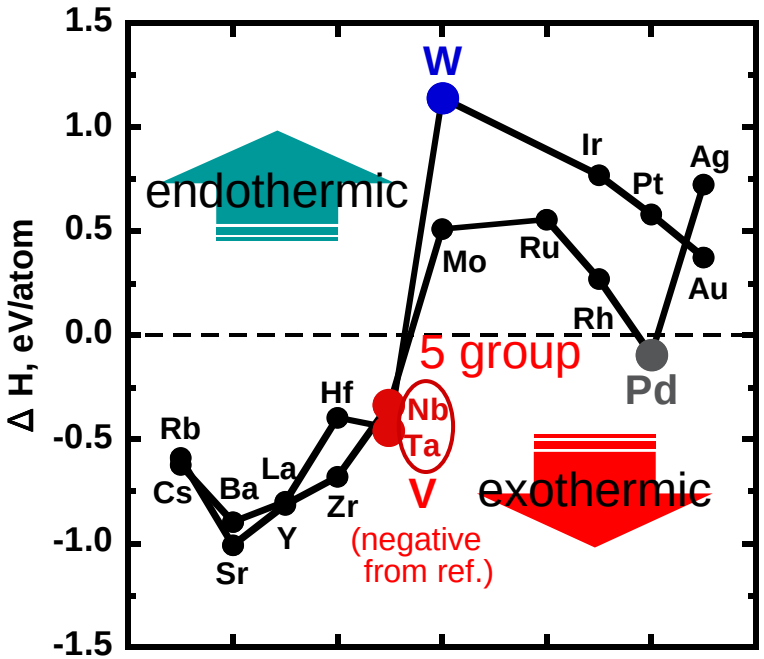


Change in the SP absorption energy with H/V in Pd-coated V.

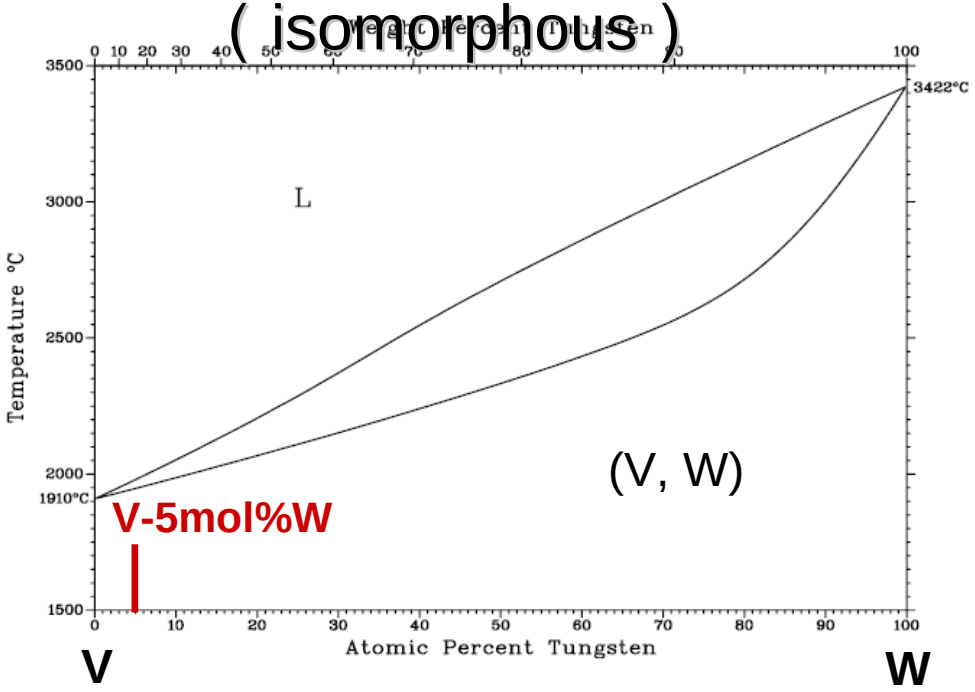
The DBTC for V is located around H/V≈0.22, and this threshold resembles the boundary for Nb.

Selection of alloying element to V

- ✓ **To suppress the dissolved hydrogen concentration**
  - ⇨ weak affinity for hydrogen
- ✓ **To make a single bcc solid solution phase**
  - ⇨ V and W (isomorphous)



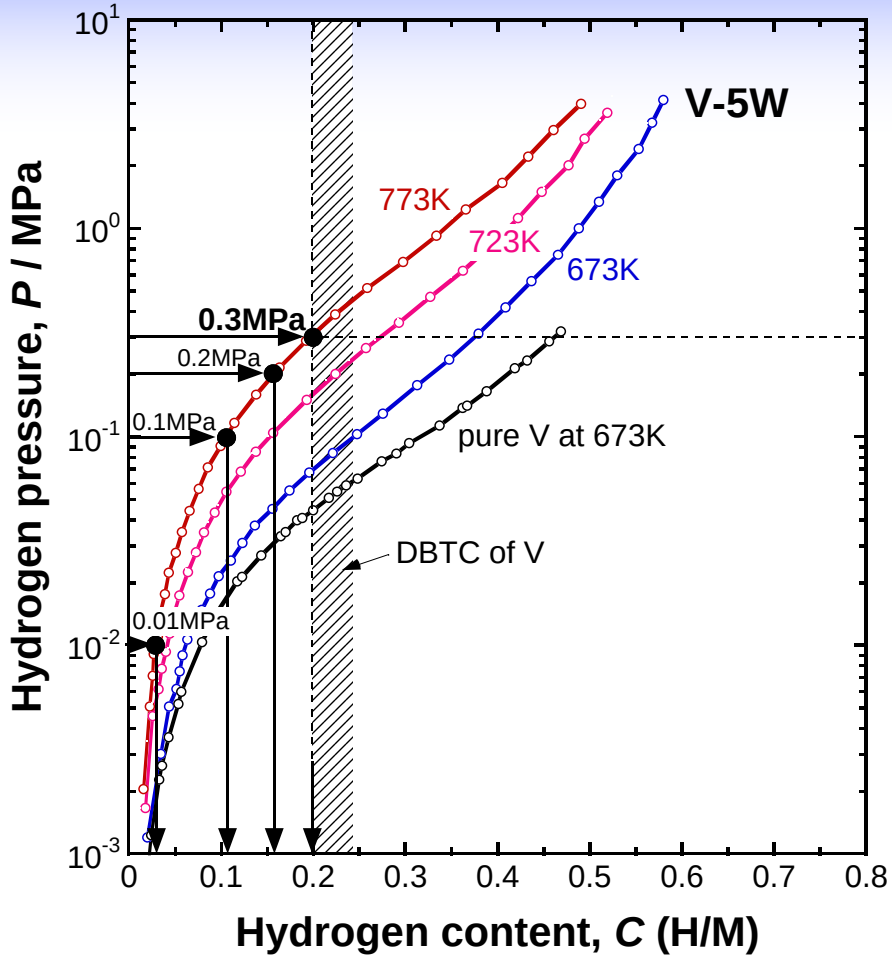
Heat of hydrogen dissolution



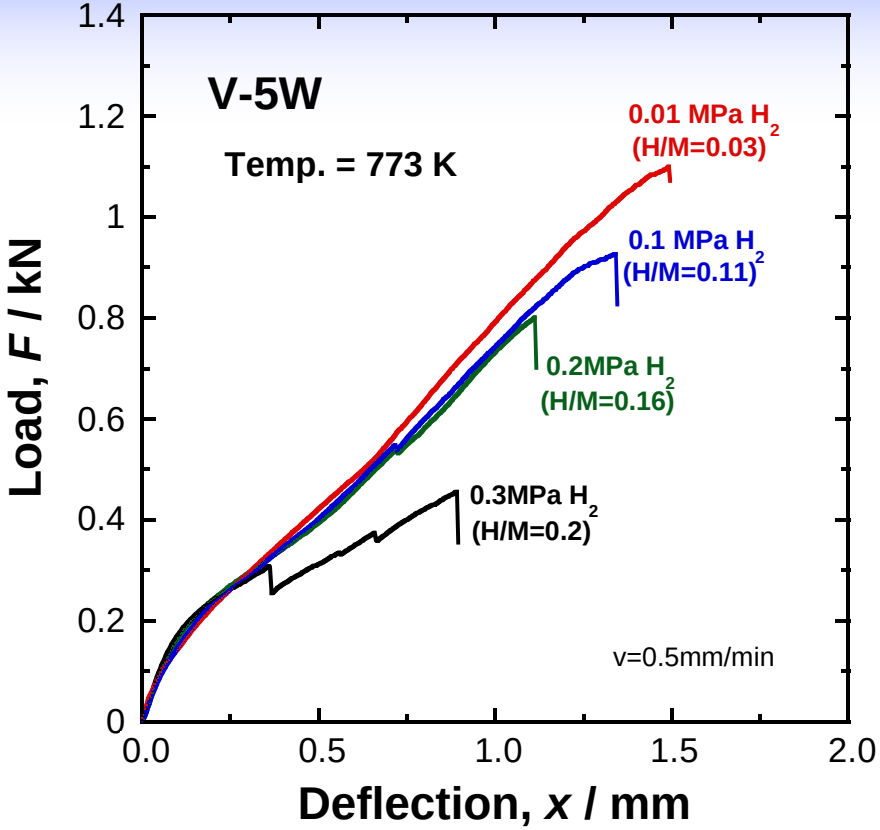
V-W equilibrium phase diagram

W was chosen as an alloying element to V.

Embrittlement avoidance by using the DBTC concept



In-situ SP test conditions of V-5mol%W alloy expressed on the PCT curves.



Load-deflection curves of Pd-coated V-5mol%W alloys measured at 773K.

The load-deflection curve slightly changes by hydrogen content, H/M, however, the high ductility of each alloy is maintained.

Nb- and V-based alloy membranes with high hydrogen permeability and strong resistance to hydrogen embrittlement can be designed and developed by using the DBTC evaluation.

Summary

- The hydrogen embrittlement of pure niobium, pure vanadium and V-5mol%W alloy membranes have been investigated quantitatively under the hydrogen atmospheres by using a newly developed *in-situ* SP test apparatus.

- The ductile-to-brittle transition hydrogen concentrations (DBTC) of them are evaluated from SP absorption energy changes between samples tested at various hydrogen dissolved conditions.

Thank you for your attention!