Detection for Nuclear Products in Palladium Deuteride/Hydride in the Evacuated Chamber

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JCF2 Meeting, Hokkaido University
October 22, 2000

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I. Introduction

o Controlled out-diffusion of deuterium from Pd in evacuated chamber.

$\Longrightarrow$

- detected T, ^{4}He, charged particles.
- observed excess heat.

♥ Advantages.

We can

→ measure the composition of the gas and the temperature together in real time.

→ reduce the background.

→ keep the surface of the sample clean.

→ SIMS analysis

♦ **Goal of this experiment**

· Reconfirmation of the results in the previous experiment.

· Understanding the nuclear reaction by detection of the nuclear products on the sample.

II. Experiment

0. Preparation of the sample.

- Cut Pd plates. (99.95%, pure)

  $12.5 \times 25.0 \times 0.3mm$

  $12.5 \times 12.5 \times 0.3mm$

- Wash with acetone and aqua resia (10sec).

- Coat a side of the sample with MnOx film
  ($\sim$ 20 nm thickness) by sputtering.

< surface analysis (SIMS)

- Load deuterium/hydrogen gas.

  $\sim$ 5 atm, $\sim$ 48hrs.

  $\Longrightarrow \frac{(D,H)}{Pd} \sim 0.7$

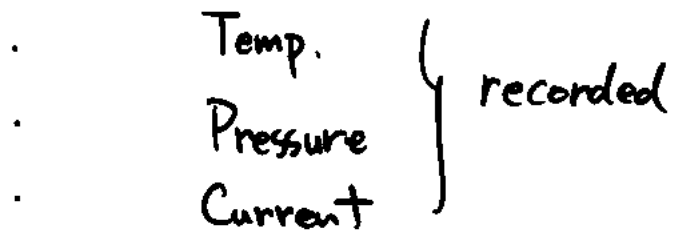
1. Set the Pd sample into the vacuum chamber.

   · stainless steel (880 cm^3)

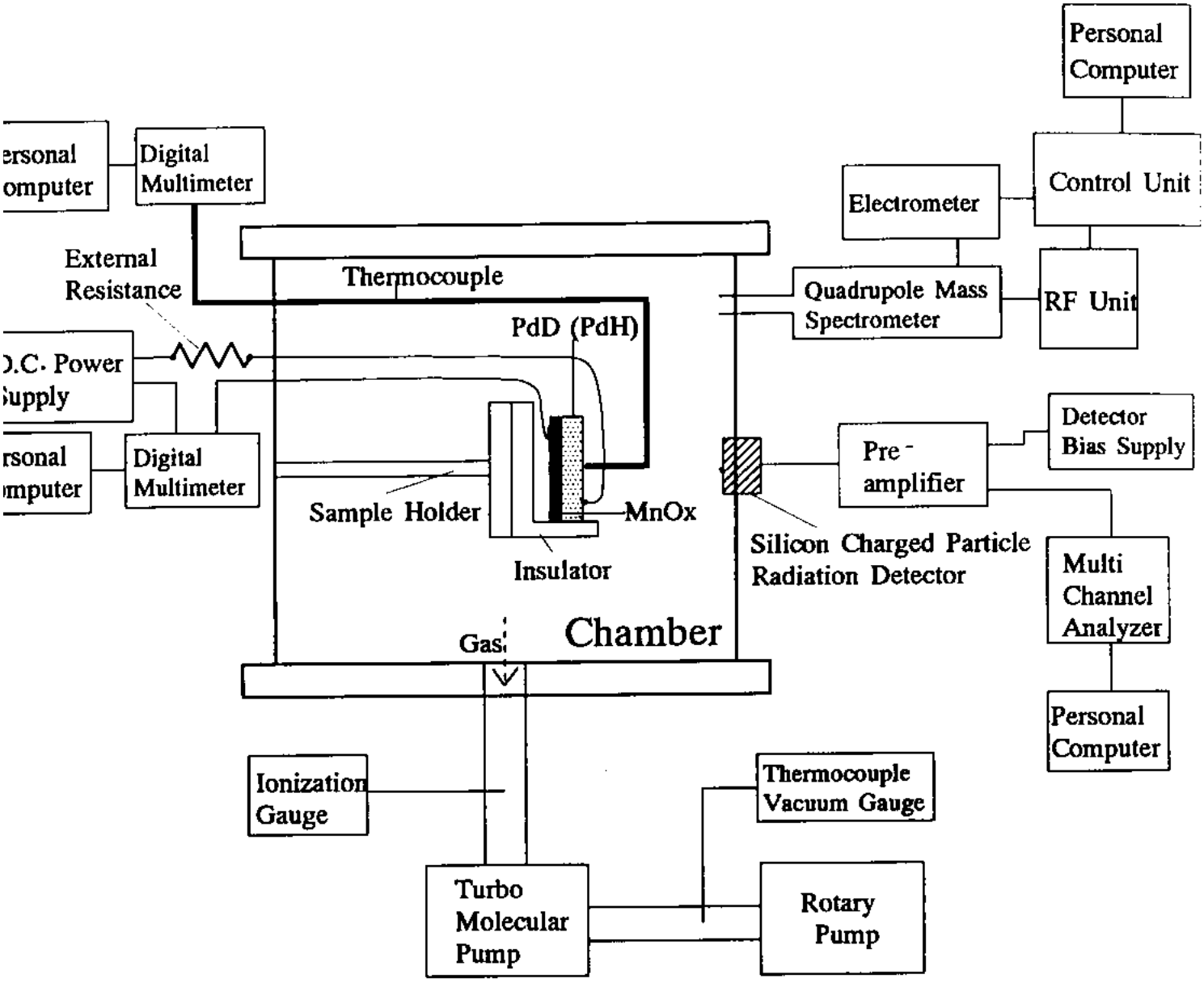
   · pumping to $\sim 10^{-4}$ Pa

2. Start to measure
   - temperature of the Pd surface by thermocouple
   - mass spectroscopy of the gas in the chamber by QMAS

3. Supply DC current.
   $\sim$ 4.0 A, $\sim$ 3 hrs.



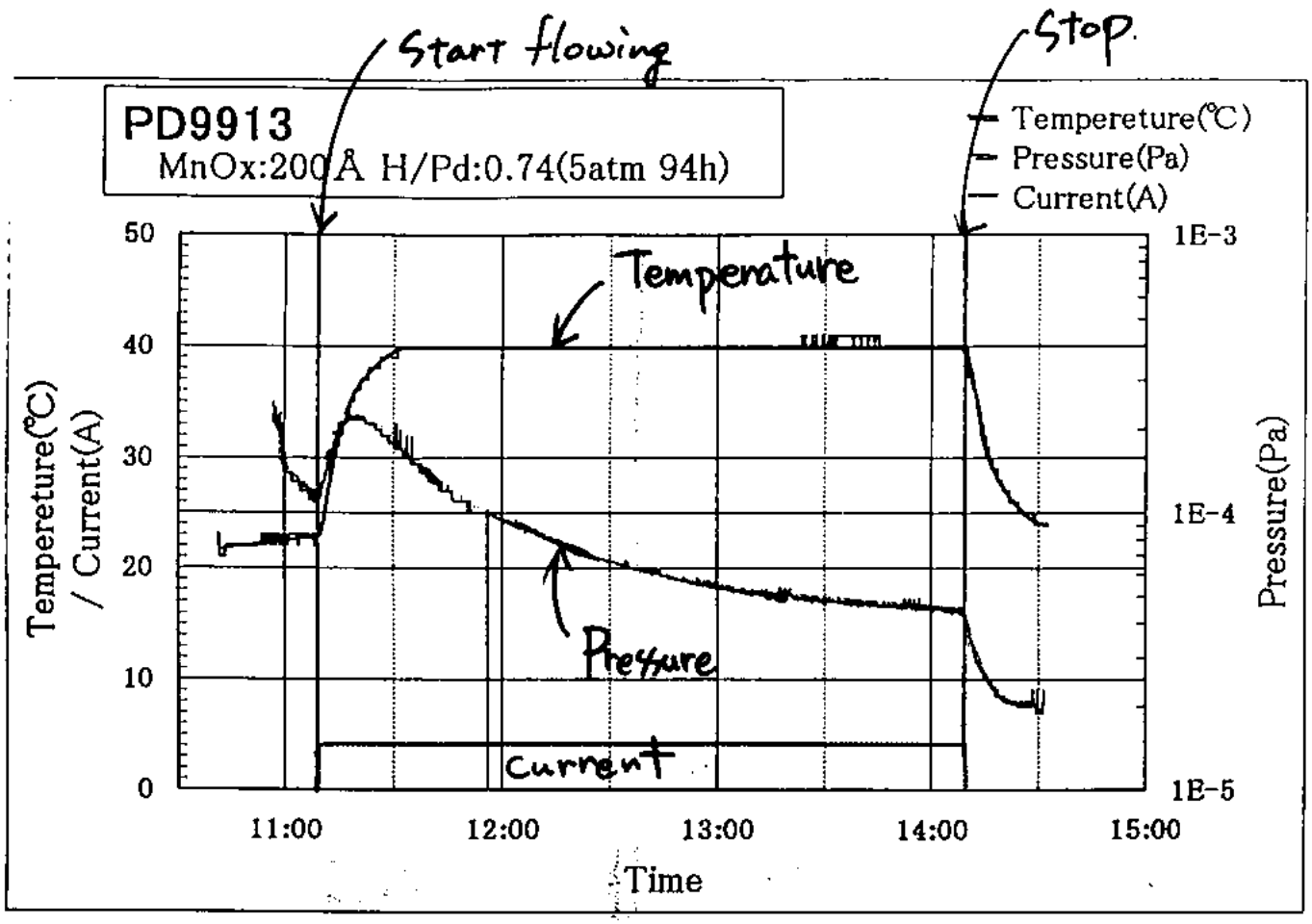
4. Stop flowing current, then look at the products on the surface of Pd.
   $\rightarrow$ autoradiography
   $\rightarrow$ Secondary Ion Mass Spectroscopy (SIMS)



Experimental setup

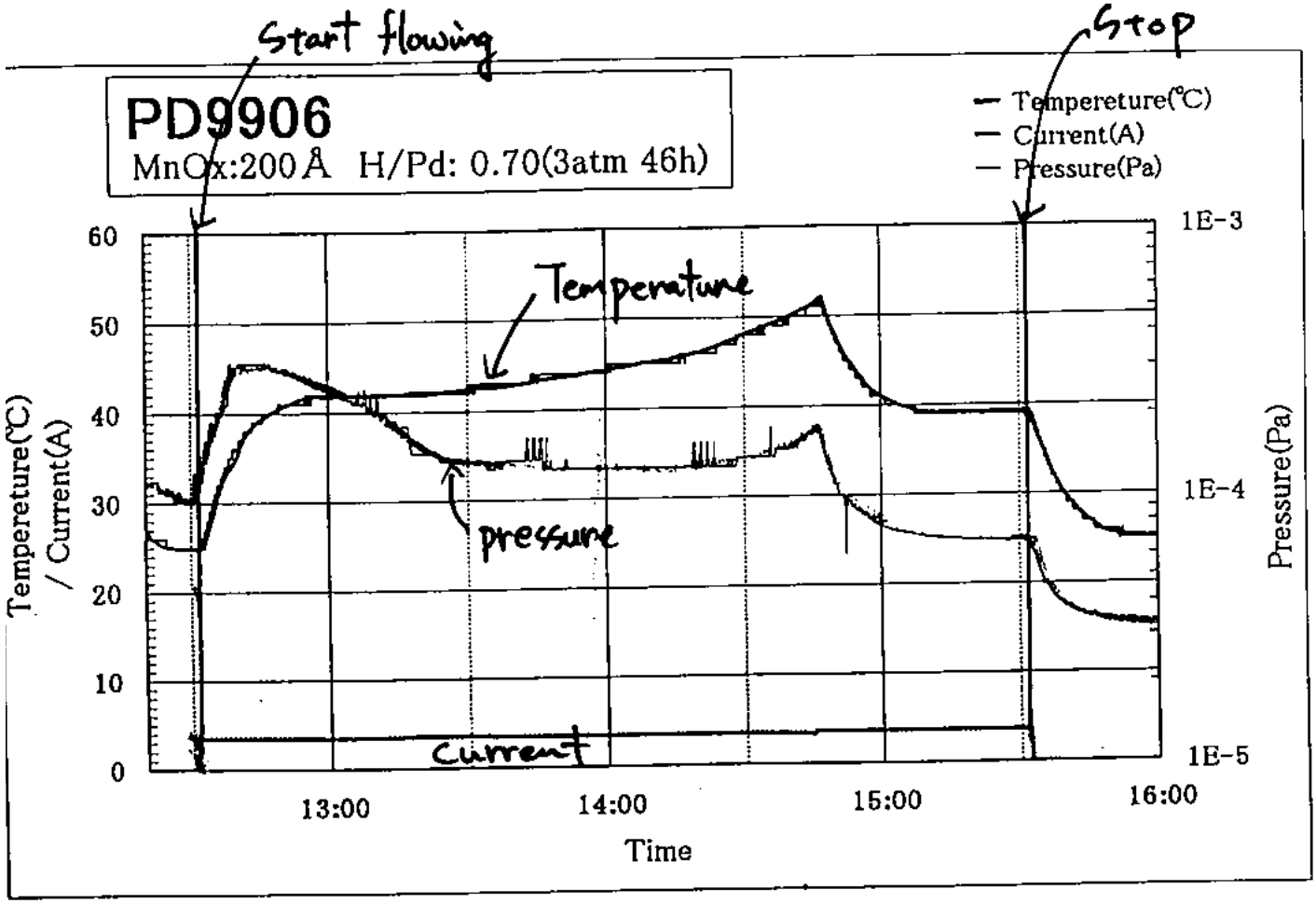
III. Results

o Time behavior of temperature and pressure for Pd deutride.



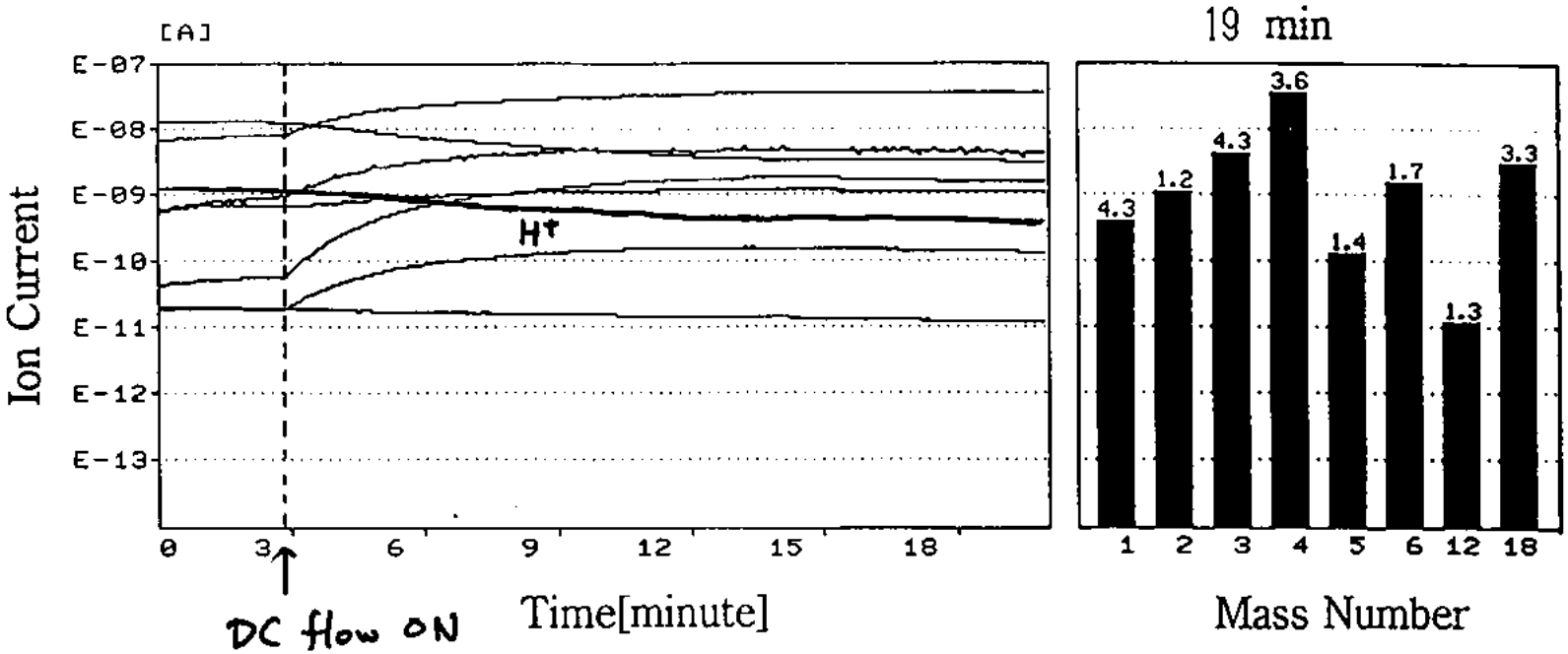
• Time behavior of temperature and pressure (cont'd)

anomalous time behavior



· SOME REACTIONS HAPPENED.

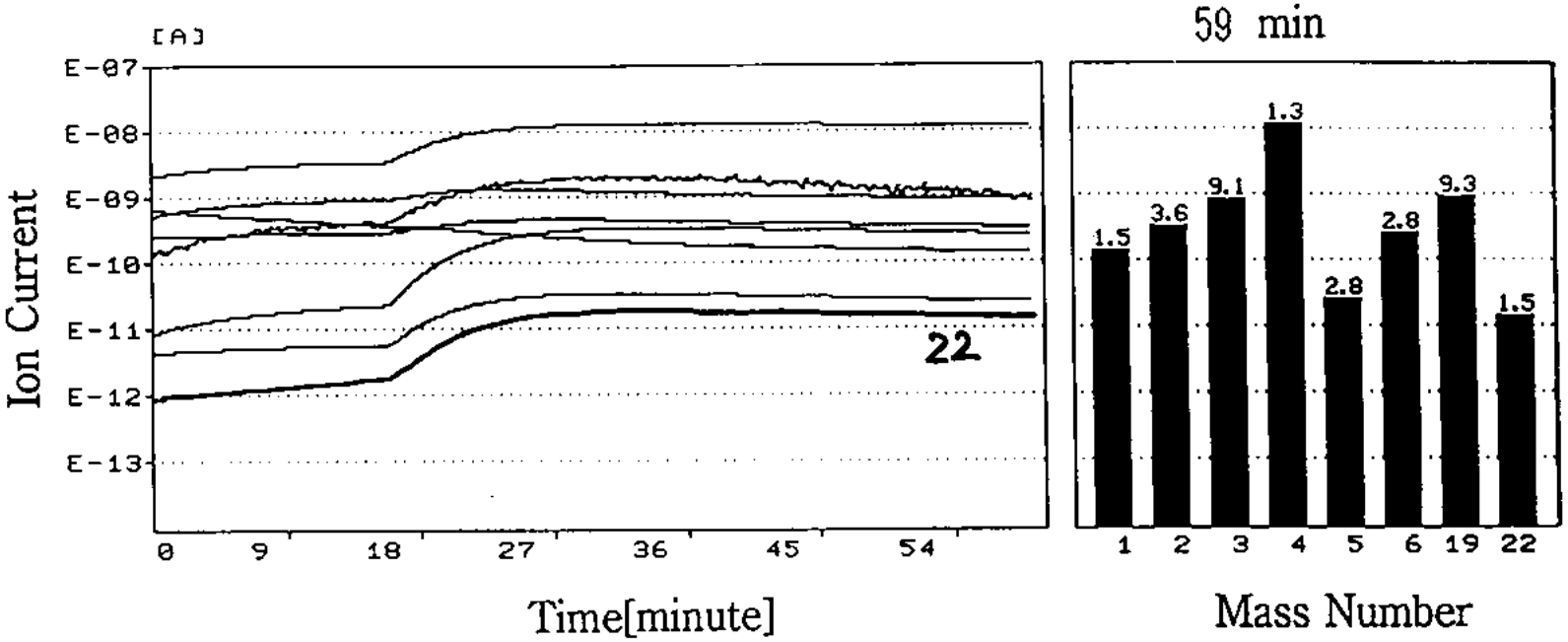
mass spectroscopy by QMAS. Pd deutride.



- 2 - 6 Increase
- No H from Pd

D, T/^{3}He, D_2/^{4}He, DT/T_2

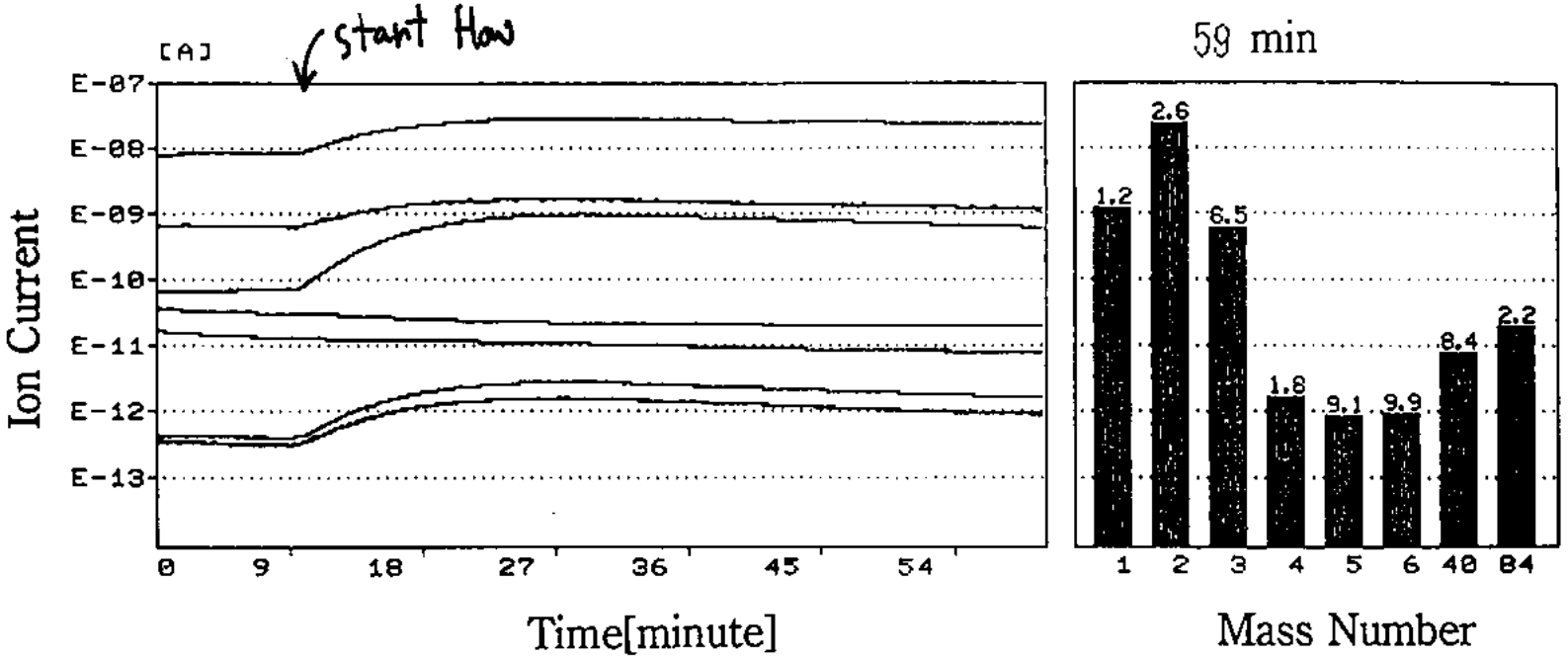
mass spectroscopy by QMAS. (cont'd) Pd deuteride



- 22 increase.

T_2O from Pd produced T.

mass spectroscopy by QMAS. (cont'd) Pd hydride.



- 1, 2, 3, 4, 6 : increase from Pd.

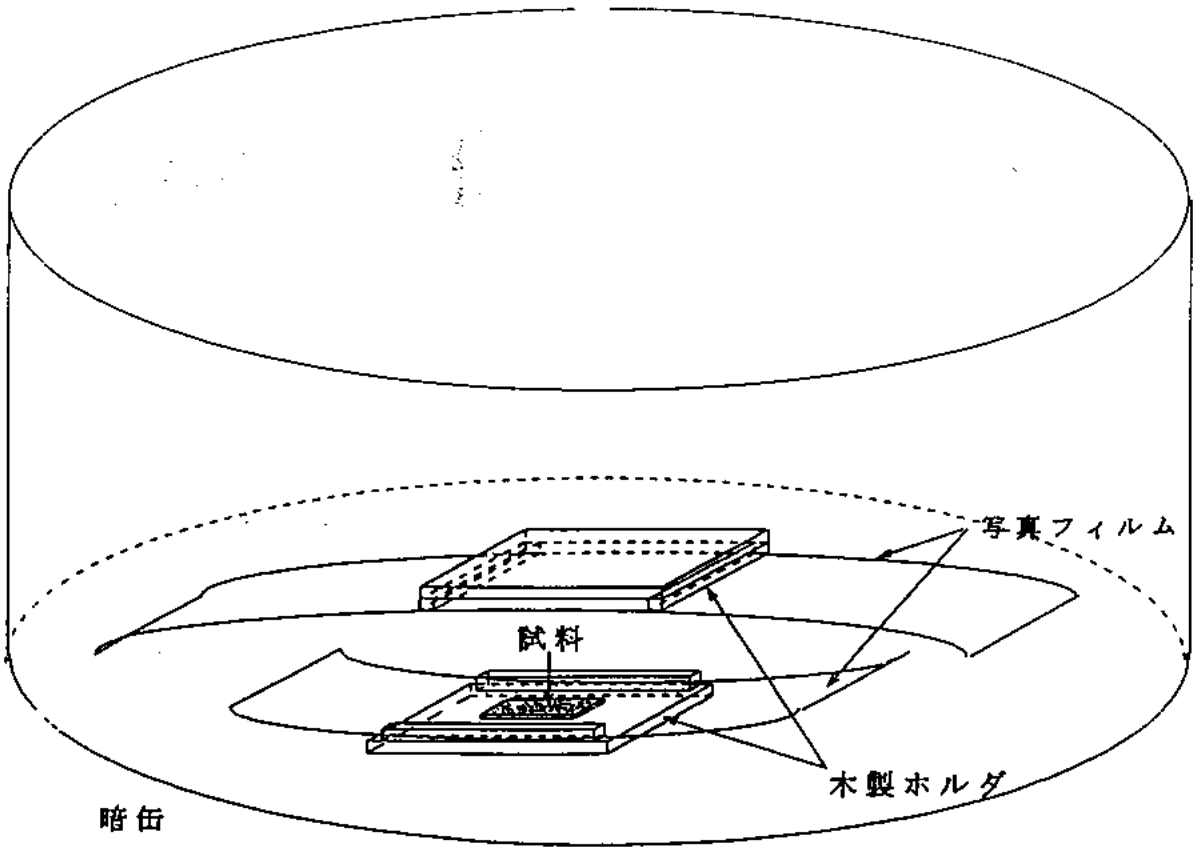
3 : HD, T,

4 : D_2, HT

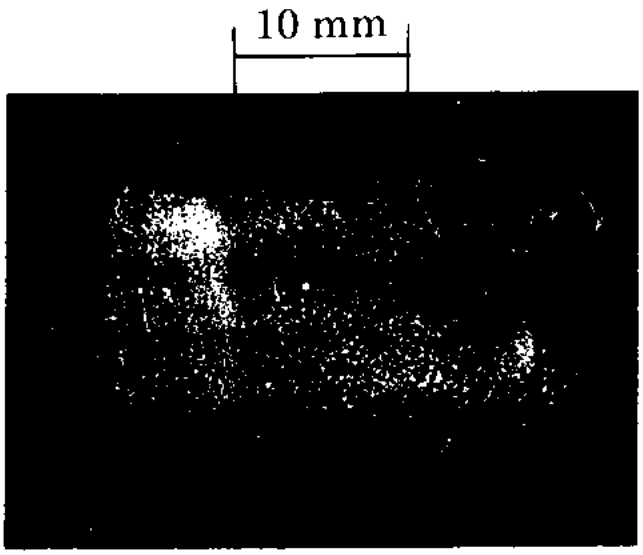
possibility of T production

○ Autoradiography

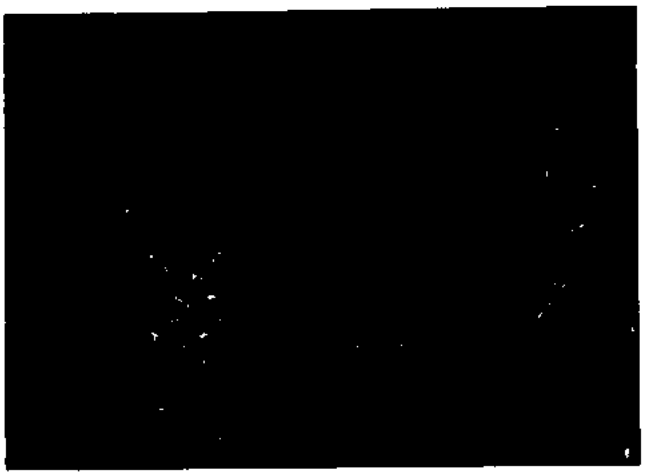
- High sensitive monochromatic film was used to detect the radiations from the Pd. (Fuji . NEOPAN. 400 PRESTO)
- Advantages:
  Free from an electric noise. Cheap. ...



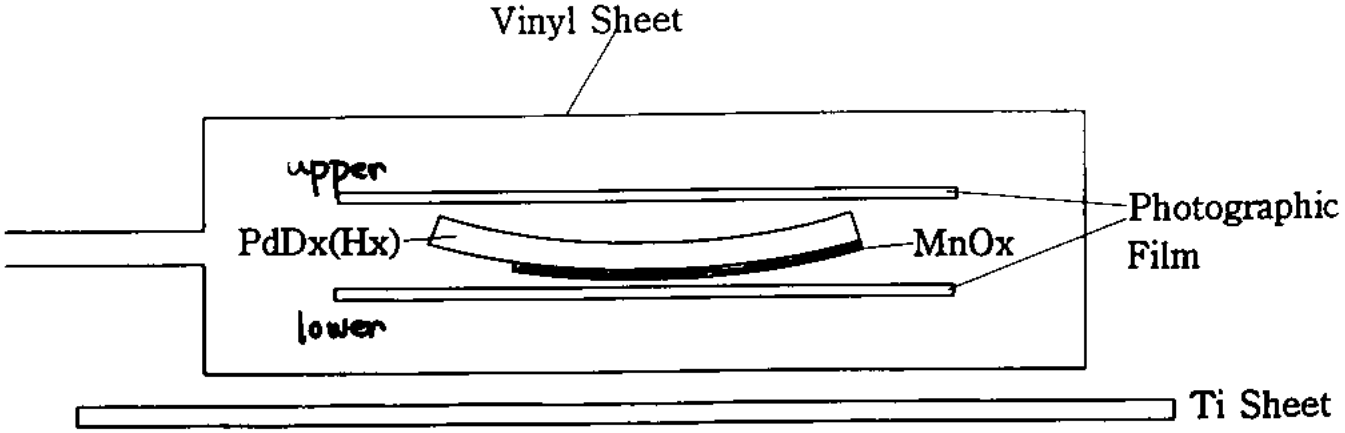
A Pair of autoradiographs after out-diffusion experiment for a Pd hydride



(a) Upper Film

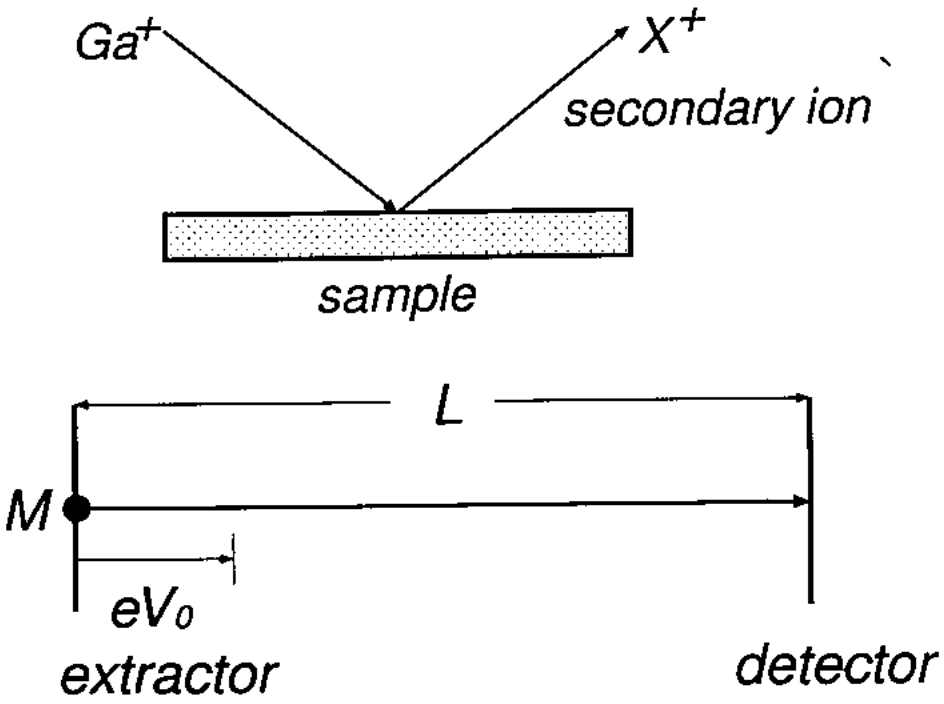


(b) Lower Film



- Investigation for the surface of the sample

Time-of-Flight Secondary Mass Ion Spectrometer (TOF-SIMS)



kinematic energy:

$$E = eV_0 = \tfrac{1}{2}Mv^2$$

$$\Rightarrow \quad t = \frac{L}{v} = L\sqrt{\frac{M}{2eV_0}}$$

- high sensitivity and good mass resolution.

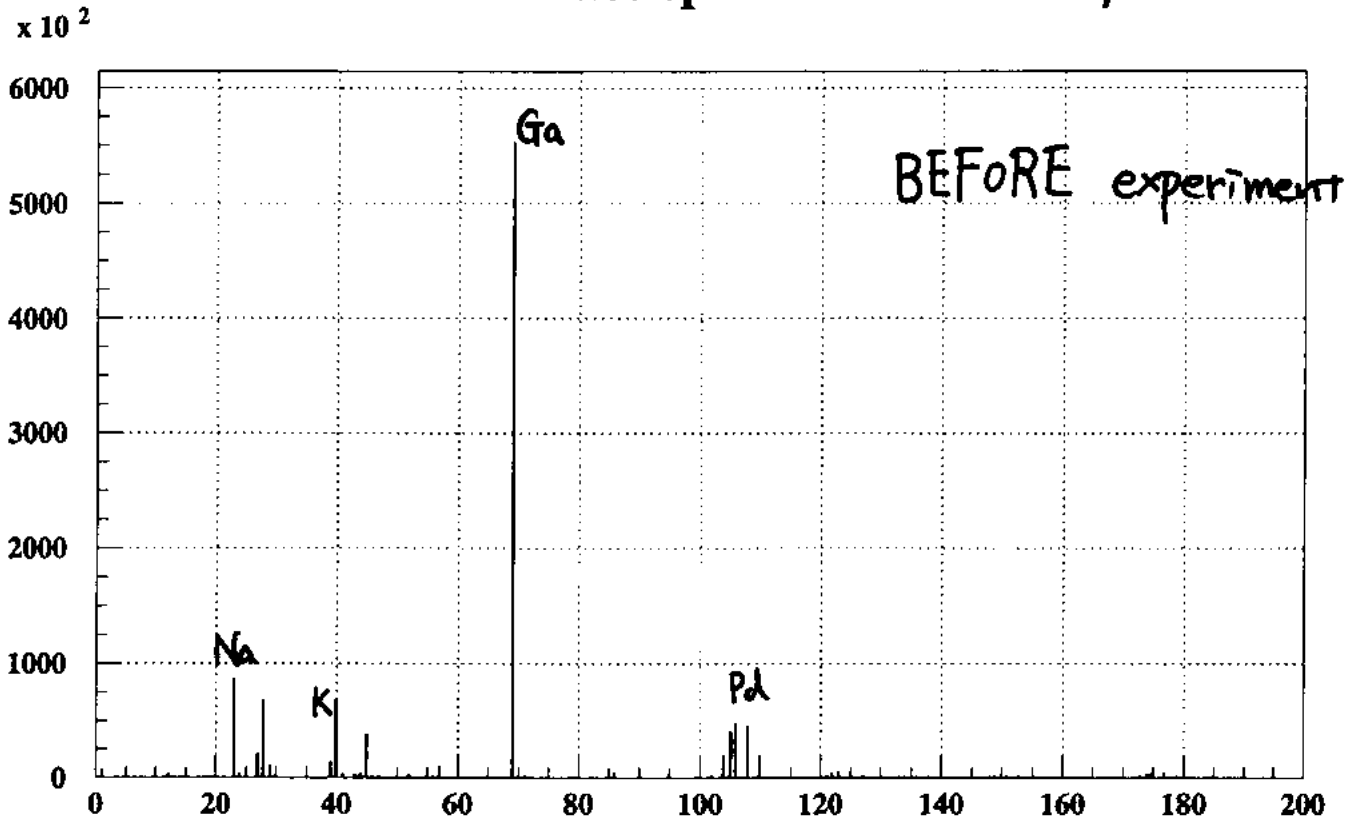
▷ *SIMS analysis*

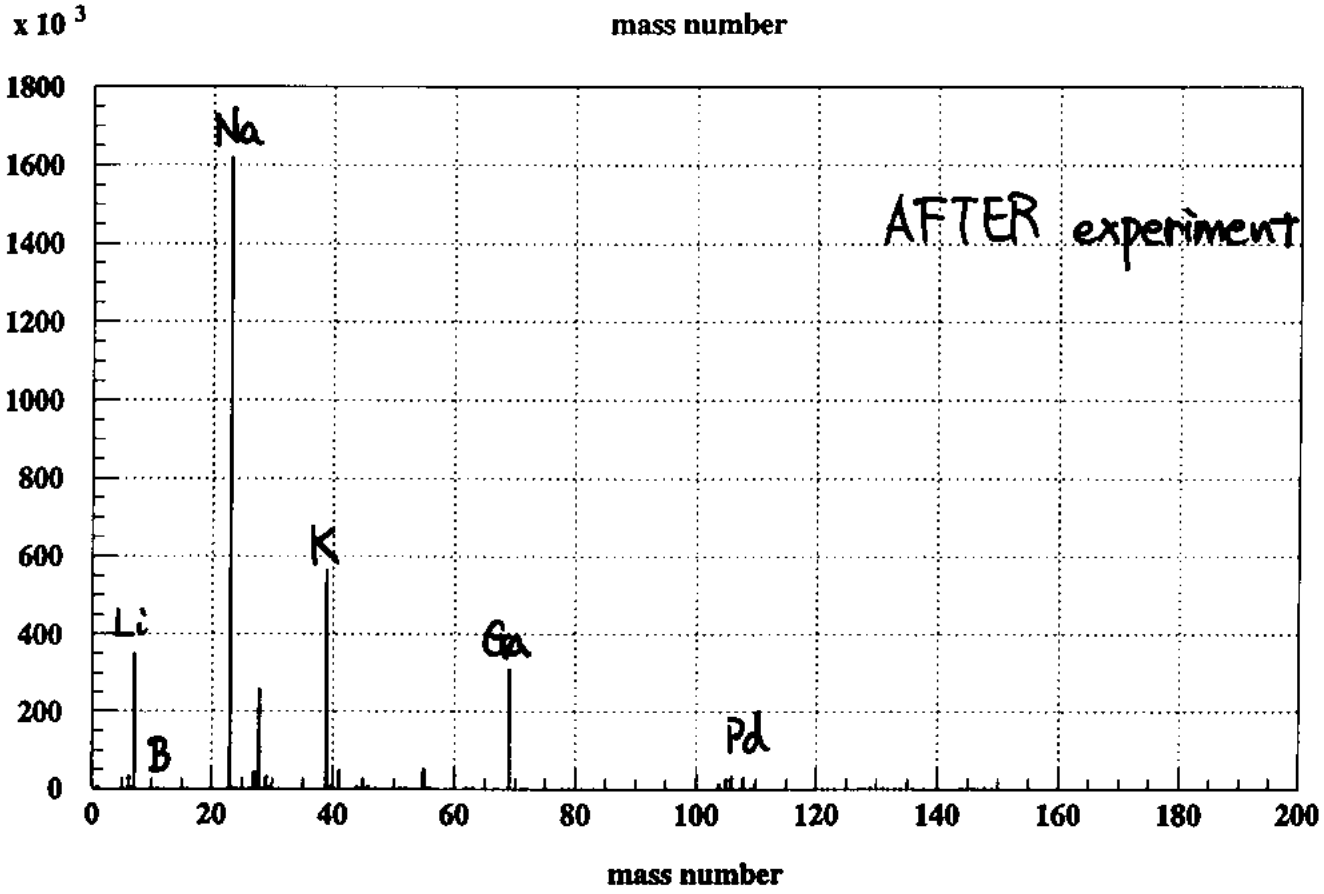
- SIMS analysis was performed for Pd hydrides BEFORE and AFTER experiments.
- Several areas ($40\mu m \times 40\mu m$) were chosen randomly on a sample and investigated.
- Each area was cleaned with sputtering (10 sec) before the analysis. $(120\mu m \times 120\mu m)$

⇓

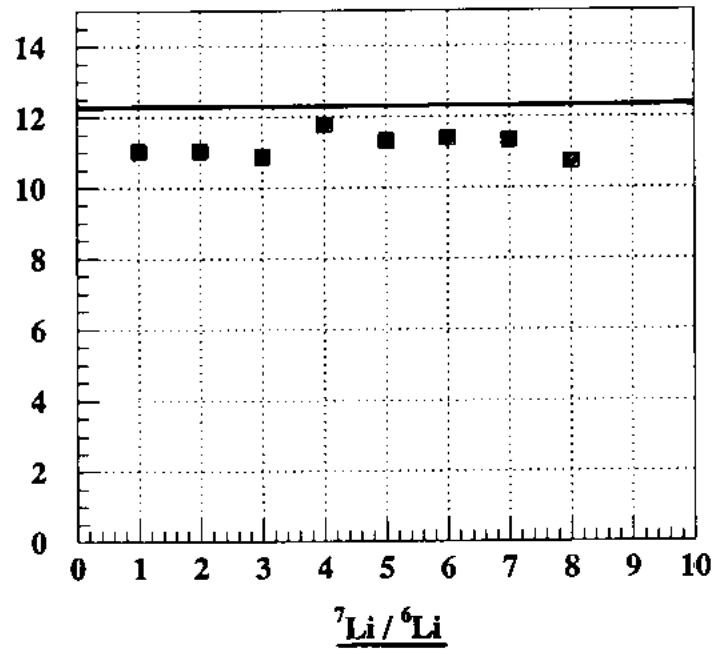
- ○ Li, B (,Fe) were detected after experiment.
  - Not detected before experiment.
  - The isotopic abundance is the same as one in the nature.
- ○ Significant increase of Na, K.
  - To discuss strictly about the quantity of the element, we need to understand details of the SIMS mechanism. (number of induced primary ion, efficiency of the ionization, ...) $N' = \underset{\sim}{\varepsilon} N$
  - We tried two normalization methods.
    → large increase for both methods.
  - Still consider the possibility of the contamination from the environment.

mass spectrum Pd hydride

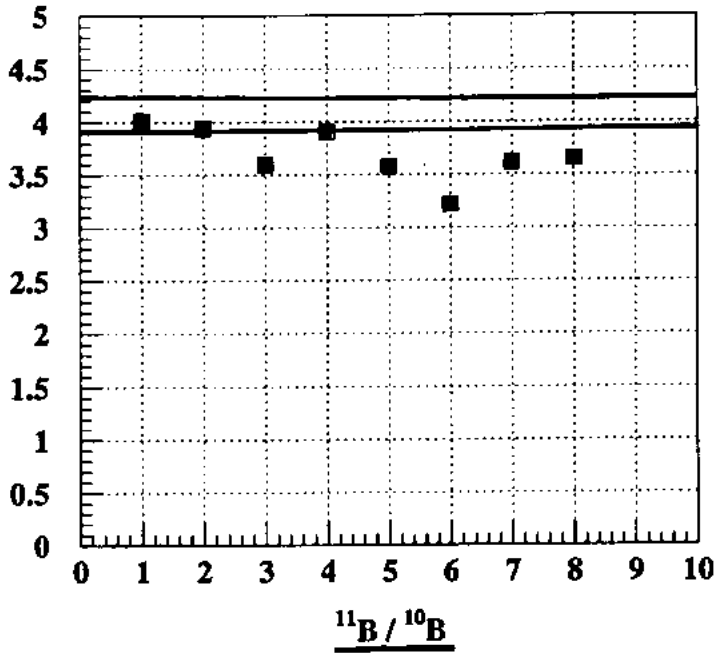




ISOTOPIC ABUNDANCE

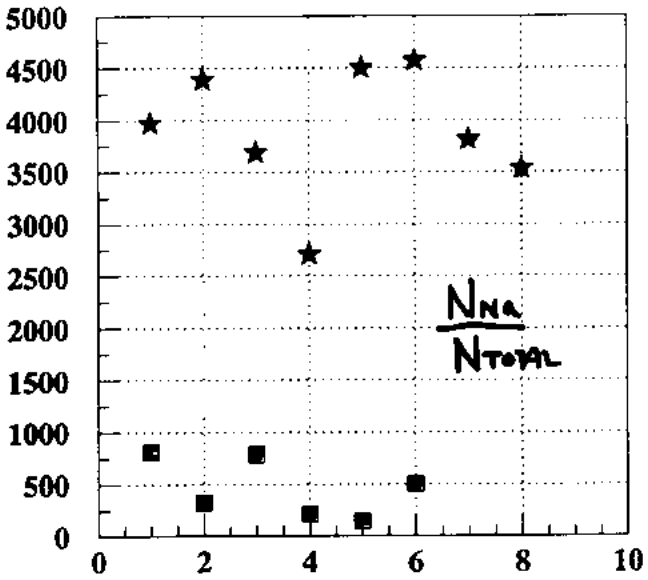


natural abundance

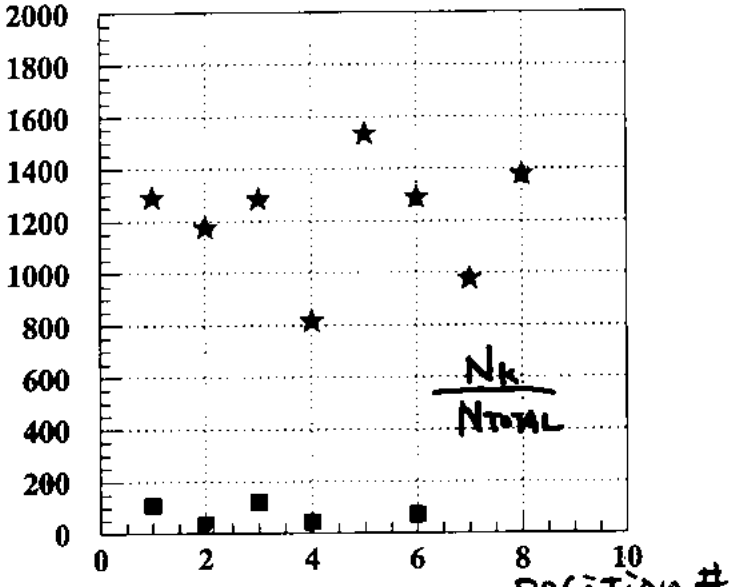


natural abundance

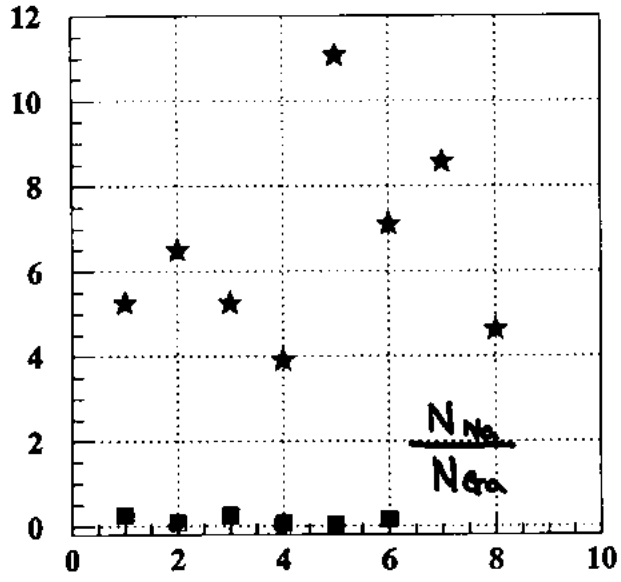
DID Na.K REALLY INCREASE?



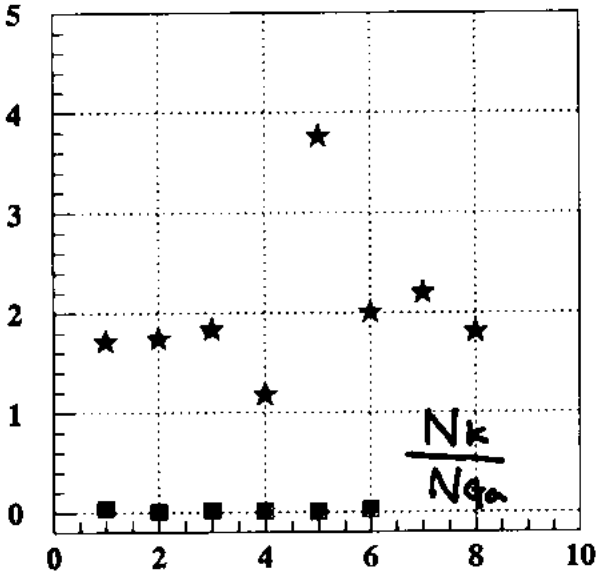
Na (normalized by total counts)



K (normalized by total counts)



Na (divide by Ga counts)



K (divide by Ga counts)

IV. Summary

- We have performed the controlled out-diffusion experiment in the evacuated chamber for Pd deuteride/hydride.

- Anomaly in time behavior of the temperature and the pressure in the chamber was observed for a few samples. (but no reproducibility ...)

- Time resolved mass spectra showed the possibility of T production.

- Radiation from the Pd was observed by autoradiography.

- SIMS analysis.
  - Li and B were detected for the Pd hydride after experiment.
  - Significant increase of Na and K components was seen.
  - Considering the possible impurities and their sources.
  - Need to understand the characteristics of SIMS.