CHEMISTRY LETTERS, pp. 2197-2200, 1989.

An Approach to the Cold Fusion through Hydrogen Isotopes Analysis by the Heavy Ion Rutherford Scattering

Minoru YANOKURA, Masaki MINAMI, Sadamu YAMAGATA, Seiichiro NAKABAYASHI,
Michi ARATANI, Akira KIRA, and Isao TANIHATA
RIKEN Insutitute, Wako, Saitama 351-01

Cold fusion phenomenon becomes an exciting topic[1,2] in this year. Fusion reaction is suggested to take place in heavy water electrolysis with a combination of platinum and palladium electrodes. To ascertain this cold fusion process, we have analyzed hydrogen and helium isotopes in the palladium electrode by means of heavy ion Rutherford scattering and got some results on material aspect of this problem.

Samples which were pre-treated by two different methods, were studied. One set of electrodes was etched by conc. sulfaric acid before electrolysis(A-group), and the other was pre-loaded with D_2 gas(B-group). (See next paragraph in detail).
Ion beam analysis were performed before and after the electorolysis so that the loading of deuterium by electrolysis can be examined. Also formation of tritium or helium-3 in the electrode, can be investigated by this method. Two different types of electrolytes, LiOD and $PdCl_3$, were employed. Conditions of sample's processing are listed in Table 1.

Deuterium was pre-loaded into a set of palladium samples(B-group) by a following method. First the samples were heated up to 420℃ in a vacuum. After keeping 10^{-6} torr of vacuum for two hours, D_2 gas was filled at a pressure of 1atm. into the chamber. The temperature was held at 420℃ for 1 hour then cooled down to room temperature

Table 1. Analyzed Palladium Samples

		Electrolysis					
		Before	After	Electrolysis conditions			
				Electorlytes	t/h	Currents/A	Voltage/V
Etched in sulfaric acid	A1	○					
	A2[a)]		○	$PdCl_3 + H_2O$	9	1.8 − 4	10
	A3[a)]		○	$PdCl_3 + D_2O$	9	4	10
Pre-loaded	B1	○					
	B2[a)]		○	$LiOD + D_2O$	120	1.2	5.0

a) Size of electrode is 12.5×8.3×0.8 mm.

slowly. These samples were kept in heavy water (D_2O), all the time after the loading.

Hydrogen isotopes in the palladium electrodes were characterized by Rutherford scattering at RIKEN heavy ion linear accelerator (RILAC). Argon ion of 61 MeV is used as an incident particle. Samples were placed at center of the scattering chamber and were irradiated by the beam with an angle of 30 degrees to the surface of sample. We eliminated scattered heavy ions using a 15 μm aluminium absorber in front of a solid state detector. The pulse height distribution of recoiled ions was accumlated. In our hydrogen analysis,[3] we can obtain not only content but also depth profile from the surface to 0.7 μm depth in palladium. Then, absolute values of hydrogen and deuterium contents have been calculated.

[A-group] Results of hydrogen analysis are shown in Fig. 1. All of A-group samples have showed only hydrogen peak, lacking a deuterium peak. Only surface hydrogen peak was observed in the sample A1. After electrolysis, A2 shows a large amount of hydrogen and A3 shows a small amount of hydrogen, not deuterium, in the palladium metal. The result suggests that if hydrogenated layer is formed on the palladium surface, it is difficult for deuterium to pass through the surface layer. The substitution of hydrogen by deuterium in a once formed hydrogenated layer seems to take long time. The analysis, therefore, showed that the sample has to be pre-treated in the manner effective for loading deuterium.

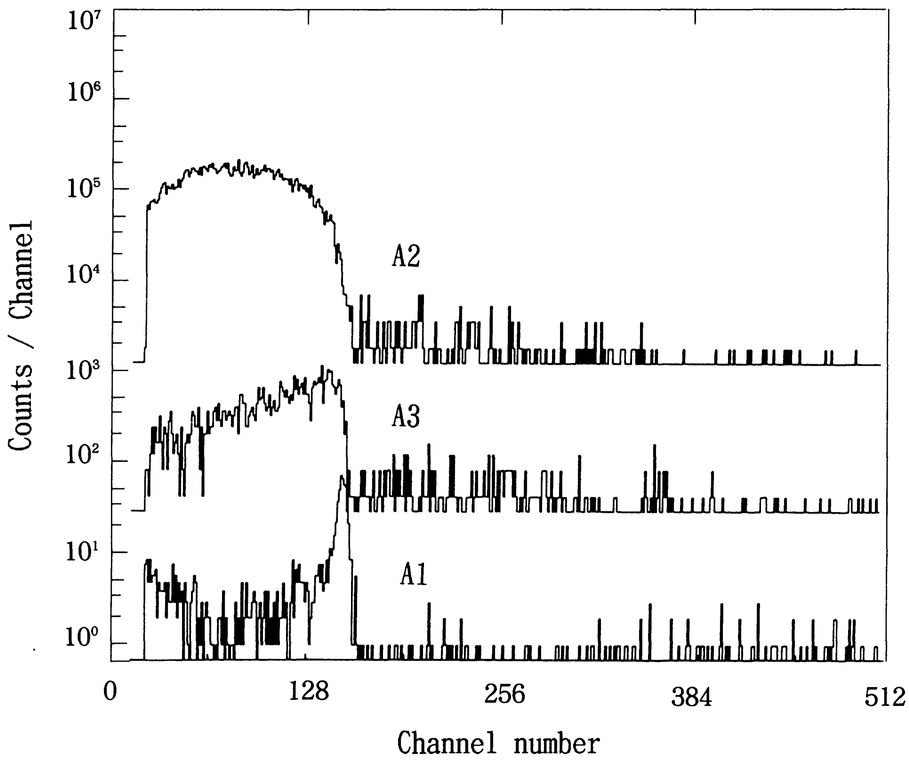

Fig. 1. Energy spectra of recoiled ions of light elements. Narrow peak of sample A1 shows surface hydrogen formed by etching.

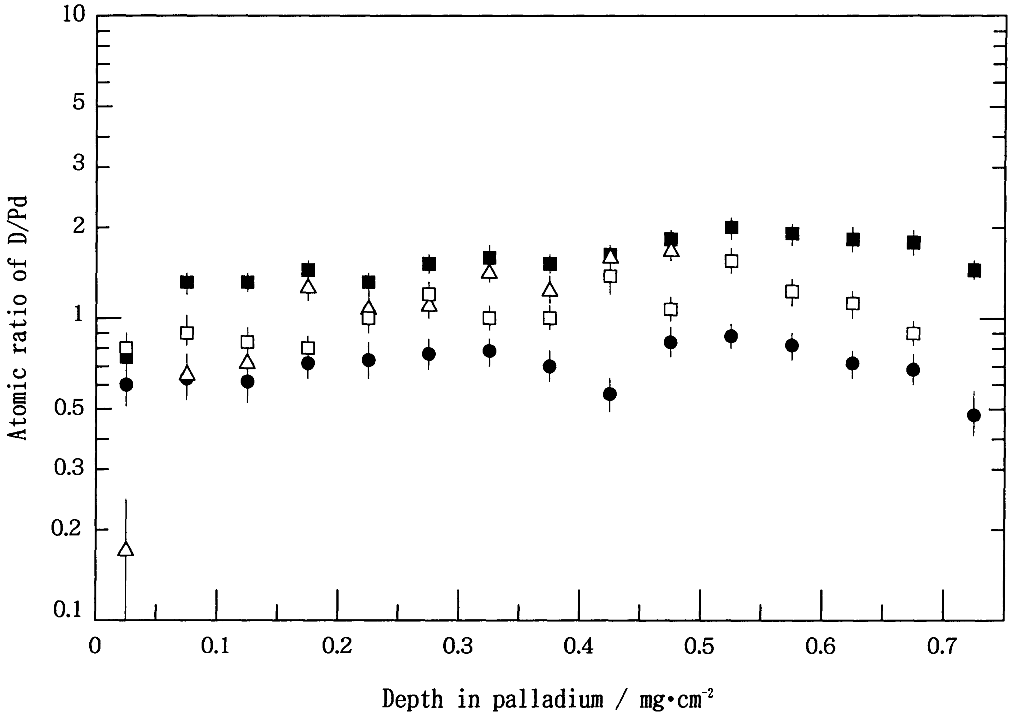

Fig. 2. Depth profile of deuterium in palladium plates.
□ B1 pre-loaded with D_2 gas before electrolysis
● B1 kept in vacuum for 1 week
■ B2 kept in heavy water after electrolysis
△ B2 kept in the atomsphere after electrolysis

[B-group] Figure 2 shows a large amount of deuterium in palladium in B1. Atomic ratio of deuterium/palladium(D/Pd) was nearly equal to 1. No hydrogen peak was observed. Repeated analyses showed that hydrogen and deuterium contents did not change under an irradiation of a few-nA 61MeV Argon ion for 1 hour. The same sample, kept in vacuum for about 1 week, was analyzed again and the atomic ratio of D/Pd has been found to decrease to 0.7 as shown in the same figure. This value showed that D/Pd atomic ratio of unity is unstable in vacuum.

The atomic ratio of D/Pd of the B2 palladium plate (after electrolysis) found to increase to 1.5. This result shows that, a large number of deuterium atoms are loaded in the palladium by electrolysis.

Furthermore, we measured differences of deuterium contents in B2 between different conditions of preservation for samples after electroysis. The sample which has been kept in the air shows that deuterium content decreases near the surface. The sample kept in heavy water, shows no significant difference in deuterium contents neither on the surface or in bulk. Between the pair of these samples, there found only differences on the surface region, but not in the bulk of palladium as shown in Fig. 2. It shows, therefore, that only deuterium located on the surface region diffused out to the atomosphere.

What we have measured is hydrogen and deuterium contents in palladium electrodes after electroysis. It is very difficult to measure hydrogen and deuterium contents during electroysis. We have no knowlege of difference of deuterium contents between during and after electroysis. It seems, however, that the obtained data reflect conditions during electolysis, because the speed of out-diffusion is observed to be small.

As a summary, if a deuterium content in palladium is important in cold fusion, our resluts showed that the pre-treatment or the surface condition of electrodes is important. Atomic ratio of D/Pd has reached to 1.5, but no significant amount of neutron has so far been detected.[4] We have also detected neither tritium nor helium-3 which would have been produced, if fusion reaction had taken place.

References

1) Electrochemically induced nuclear fusion of deuterium, M.Fleischmann, S.Pons, and M.Hawkins, J. Electroanal. Chem., 261, 301 (1989).
2) Observation of cold nuclear fusion in condensed matter, S.E.Jones, E.P.Palmer, J.B.Czirr, D.L.Decker, G.L.Jensen, J.M.Thorne, S.F.Taylor, and J.Rafelisk, Nature, 338, 737 (1989).
3) Reliability, Detection Limit and Depth Resolution of The Elastic Recoil Measurement of Hydrogen, H.Nagai, S.Hayashi, M.Aratani, T.Nozaki, M.Yanokura, and I.Kohno, Nuclear Insutruments and Methods in Physics Research, B28, 59 (1987).
4) To be published.

(Received August 19, 1989)