



Research Article

Nuclear Transmutation on a Thin Pd Film in a Gas-loading D/Pd System

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Abstract

This paper discusses the deformation and elemental distribution of different palladium film surfaces after loading and unloading many times deuterium gas in the system.

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1. Introduction

When deuterium gas permeates through a thin palladium film, it was thought as a diffusion process only. However, after about 80 times absorption and desorption processes accompanied with permeations, nuclear transmutation was discovered on the surface of palladium film using Scanning Electron Microscopy (SEM).

At first glance, it was noticed that the macroscopic deformation of palladium was very large that the palladium film might increase its thickness while decrease its diameter of a rounded palladium film. The stress at the rounded sealing line might be so strong that it even cuts the palladium film into two pieces: the central rounded piece and the ring-shape edge piece.

The SEM analysis revealed that new elements (Cu, Zn, Si, etc.) were detected in the permeation area, but there were no such elements in the original palladium film or in the ring-shape area where no deuterium permeation happened. The temperature of palladium film was much higher than that of Iwamura experiments in Advanced Technology Research Center, Mitsubishi Heavy Industries [1,2]. Besides, there was no super lattice on the surface of our palladium films. Metallography analysis will be shown as well [3].

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2. Apparatus

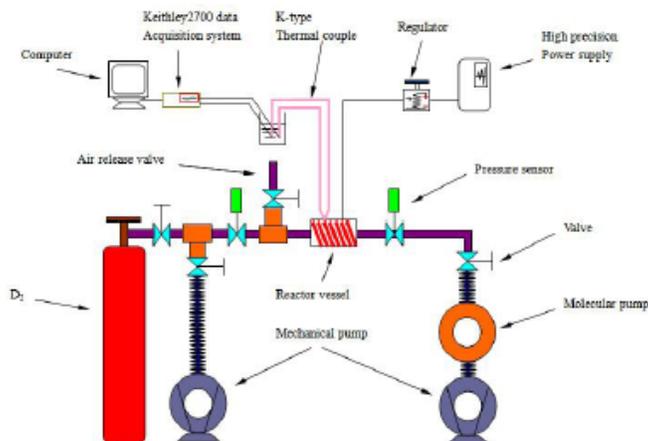


Figure 1. Deuterium gas loading palladium film system.

Figure 1 shows the sketch of deuterium gas loading palladium film system. The reactor vessel is separated by a $\phi 20 \text{ mm} \times 0.1 \text{ mm}$ palladium film into two parts. One is gas room, and the other is vacuum room. There is a 4 MPa

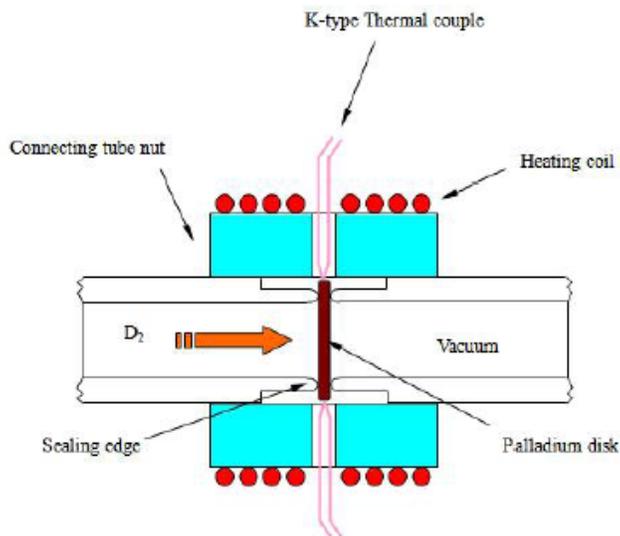


Figure 2. Palladium sealing part.

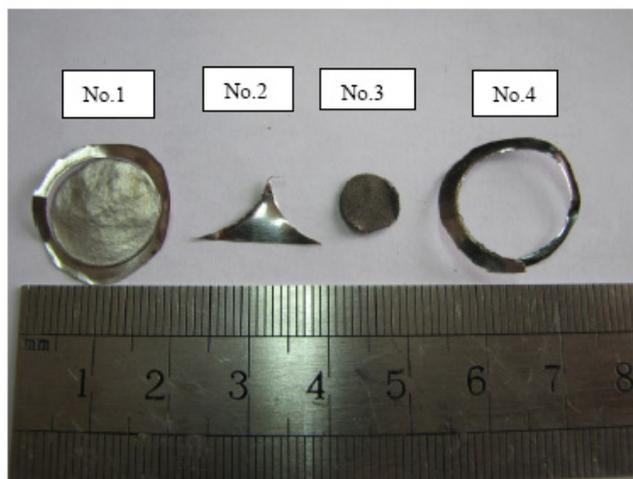


Figure 3. Palladium film samples.

D₂ gas bottle with purity of 99.5% at gas room side. Some valves are connected into pipes for checking or changing the system. When we begin to load sample gas into gas room, we have to use mechanical pump to evacuate the pipes first in order to avoiding gas pollution.

There is a molecular pump and a mechanical pump connected in series for high vacuum at vacuum room side. We set a power supply with high precision of $\pm 1\%$ in front of a voltage regulator, so we can add variable power to a heating coil which is equably wound on the swagelok sealing reactor vessel. We use K-type thermo couple to measure the temperature at some places where we think necessary. The required temperature includes the edge of Pd film, heating coil and connecting tube nut.

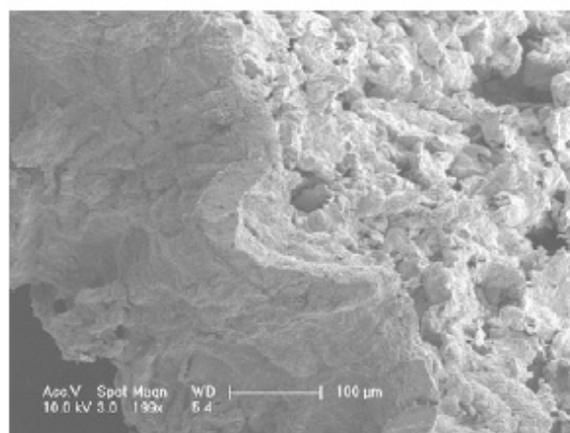


Figure 4. No. 3 sample 100 μm SEM.

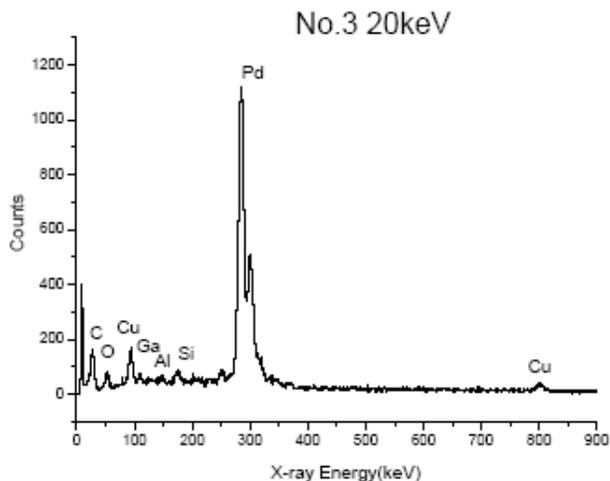


Figure 5. No. 3 sample 20 keV EDS.

Some pressure sensors are placed in pipes to monitor the gas loading pressure or vacuum. Figure 2 gives the structure of palladium film sealing part.

The palladium film is sealed by Swagelok stainless steel sealing edges when the connecting tube nut screws down conjuncting gas room and vacuum room. A round sealing mark forms at each surface of palladium film. The diameter of the mark is $\phi 16$ mm. We put the K-type thermo couple into tiny hole in the nut to measure temperature. The experimental process is described as below:

- (1) Evacuate the whole system (both gas room and vacuum room).
- (2) Stop pumping all the tubes, then load deuterium gas into gas room.
- (3) After the solution of deuterium finished, add power to heating coil to raises system temperature.

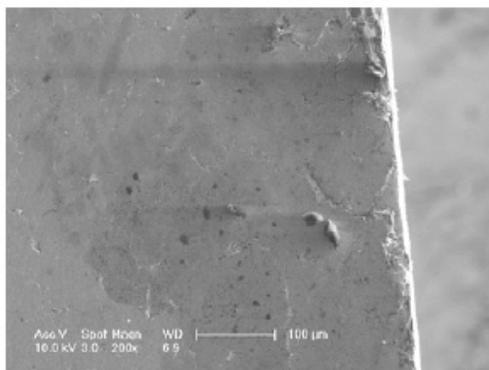


Figure 6. No. 4 sample 100 μm SEM.

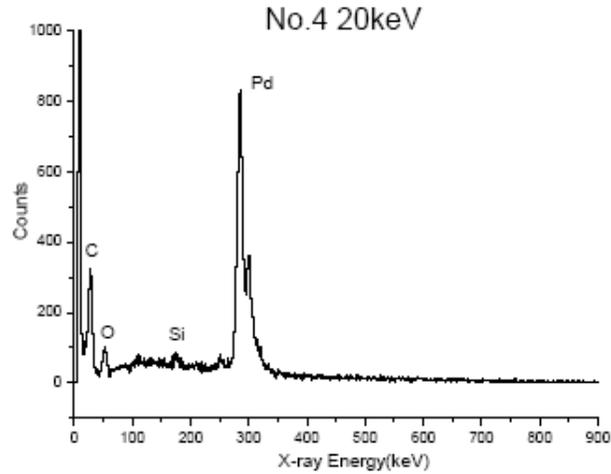


Figure 7. No. 4 sample 20 keV EDS.

- (4) After the temperature increases higher than 300°C , keep heating power unchanged and wait temperature stable [4,5].
- (5) Pumping the system with both gas room and vacuum room.
- (6) Decrease the heating power slowly until the system temperature equals the atmosphere temperature.

3. Results

Figure 3 shows the virgin sample and experimental sample. After many times deuterium gas loading and unloading, the surface of palladium film shows great change and deformation [6]. No. 1 sample has been loaded and unloaded by deuterium gas for more than 10 cycles. We can clearly see that the round area which has met deuterium gas becomes rough on surface, but the outside ring which is cut by sealing line mark keeps unchanged. No.2 sample is the virgin

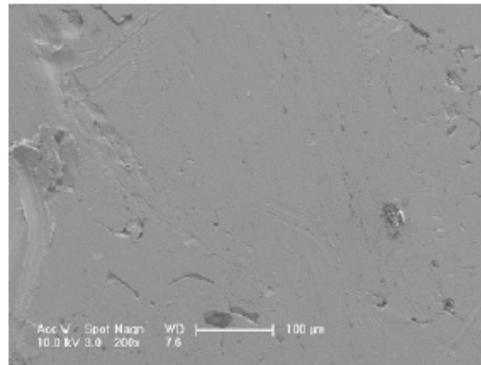


Figure 8. No. 2 sample 100 μm SEM.

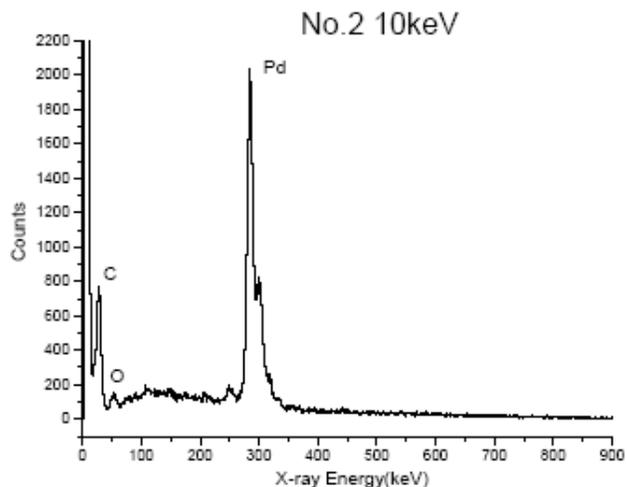


Figure 9. No. 2 sample 10 keV EDS.

palladium film. No. 3 sample has been loaded and unloaded by deuterium gas for about 80 cycles. It was cut by sealing line mark with thickness increasing and diameter decreasing. No. 4 sample is an outside ring of No.3 round piece. It nearly remains unchanged.

Figure 4 shows No. 3 sample 100 μm SEM figure. Because it suffered about 80 times deuterium gas loading, the metallic phase of palladium surface and edge shows great change. The smooth surface becomes porous and loose. A 20 keV EDS analysis gives the chemical element distribution (Fig. 5). There are two characteristic peaks of Cu distributing at both sides of Pd peak. Iwamura has found Cu many times in his research work [1,2]. Si peak is also observed in the curve.

Therefore, we are interested in No. 4 sample surface element distribution. It was separated from No. 3 sample by

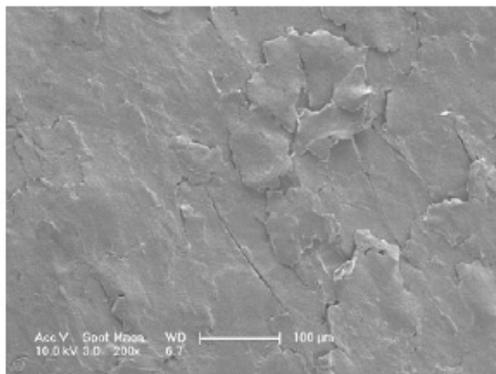


Figure 10. No. 1 sample 100 μm SEM.

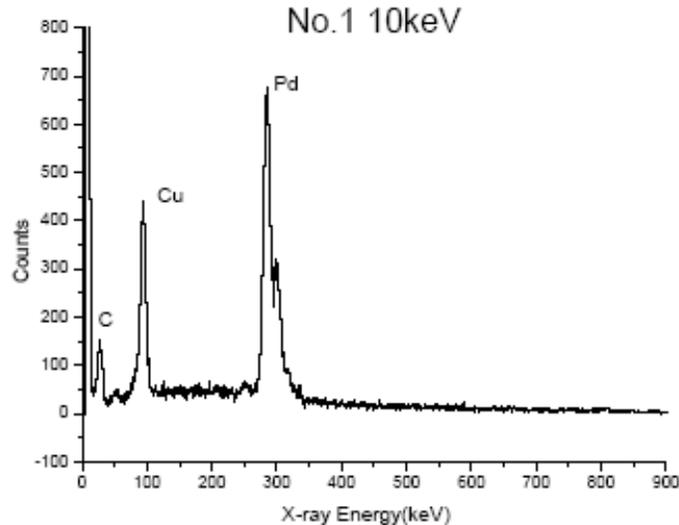


Figure 11. No. 1 sample 10 keV EDS.

sealing line mark and keeps isolated from deuterium gas. Its surface keeps smooth after the experimental cycles. The edge cut by sealing line mark is formal. We can see that in Fig. 6. Figure 7 20 keV EDS shows only Si peak besides Pd, C and O peak.

Figures 8 and 9 separately show the virgin palladium film SEM and EDS. The surface of No. 2 sample is the smoothest of all. The spectrometer curve looks like No. 4.

Figure 9 indicates that No. 2 virgin palladium film is clear. No. 4 outside ring piece shows no Cu signal on the surface but No. 3 round piece dose. Therefore, Cu may be caused by nuclear transmutation. Both on No. 3 and No. 4 sample surface we detect weak Si signal like a lot of other gas loading research groups. No. 1 sample central surface becomes loose and flaked as shown in Fig. 10. Its EDS curve also appears Cu peak seen in Fig. 11.

4. Discussion

Through SEM analysis of different palladium film and virgin sample, we study the gas loading D/Pd system. The deformation of palladium film is observed when deuterium flux generates. The element distribution of experimental palladium film surface is analyzed by X-ray EDS, and some elements (Cu, Si, etc.) show anomalous distribution. Stepped work is to find correlation between anomalous heat and element, and to eliminate noise and pollution of experiments.

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