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RELATIONSHIP BETWEEN OPEN-CIRCUIT-VOLTAGE AND HEAT PRODUCTION IN A PONS-FLEISCHMANN CELL

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INTRODUCTION

Lack of reproducibility is still the major reason CANR is not generally accepted and has not advanced into commercial use. The ability to reproduce any phenomenon depends on knowing the major variables and conditions required for the events to operate. In the case of cold fusion, even fundamental factors such as the D/Pd ratio and the crystal structure of the nuclear-active regions are not known. It is the intent of this paper to demonstrate several techniques for obtaining such information and the results obtained from their application to the Pons-Fleischmann Effect.

Reproducibility is a materials problem. The nuclear reactions only occur when the required chemical environment is created, the so-called nuclear-active-state (NAS). Some techniques and environments create these conditions more easily than others. This paper will address the electrolytic method of reacting deuterium with palladium.

Considerable experience now demonstrates that a critical D/Pd ratio is required for anomalous energy to be produced. It is also clear that this critical composition first occurs in the near-surface region of the Pd cathode. The composition that can be reached in this region depends on a number of factors including surface barriers and surface penetrating cracks. I would like to discuss several methods used to investigate the characteristics of the surface region and the understanding obtained therefrom. In addition, I would like to show the results of a direct measurement of the near-surface composition.

The average bulk composition can be measured in several ways. However, this quantity is only indirectly related to the near-surface composition where the nuclear reactions actually occur. The problem is to determine the surface composition more directly. This can be done in a relative way using the open-circuit-voltage (OCV), which is proportional to the Gibbs Energy of deuterium. While the exact relationship between Gibbs Energy and composition is not known, especially in the high deuterium region, the relative behavior of the OCV is useful in understanding the behavior of the surface. An approximate value for this composition also can be measured directly using thin films of palladium deposited on an inert substrate.

Another useful method is the loading efficiency. This quantity is sensitive to the presence of surface barriers, hence can quickly identify material which is <u>not</u> capable of achieving high compositions.

The final method uses the deloading behavior. This quantity is sensitive to the presence of cracks as well as to surface impurities which affect the ability of deuterium to leave the sample. The deloading rate used in this paper is obtained by taking the slope of D/Pd vs time.

The rate of gain competing with the rate of loss determines the ultimate limiting composition. Therefore, factors that affect both sides of the equation must be understood. Techniques for measuring each variable will be discussed.

This work is described in more detail in a paper soon to be published in J. Alloys and Compounds.

APPARATUS

The samples are electrolyzed in a Pyrex cell (Fig. 1) held at constant temperature by a surrounding jacket through which water flows from a constant-temperature bath. The OCV is measured between the cathode and an external platinum metal gauze, through a Luggin capillary terminating within 1 mm of the surface. Even though the reference electrode is not bathed in hydrogen, it is sufficiently stable for the purpose of this study. A second capillary is located on the other side of the sample such that the average behavior of the cathode surface is measured. Measurements are made while the circuit is opened for 1 to 5 sec. The cell also contains a recombiner catalyst. Graphite impregnated with Pt is used as a catalyst when the
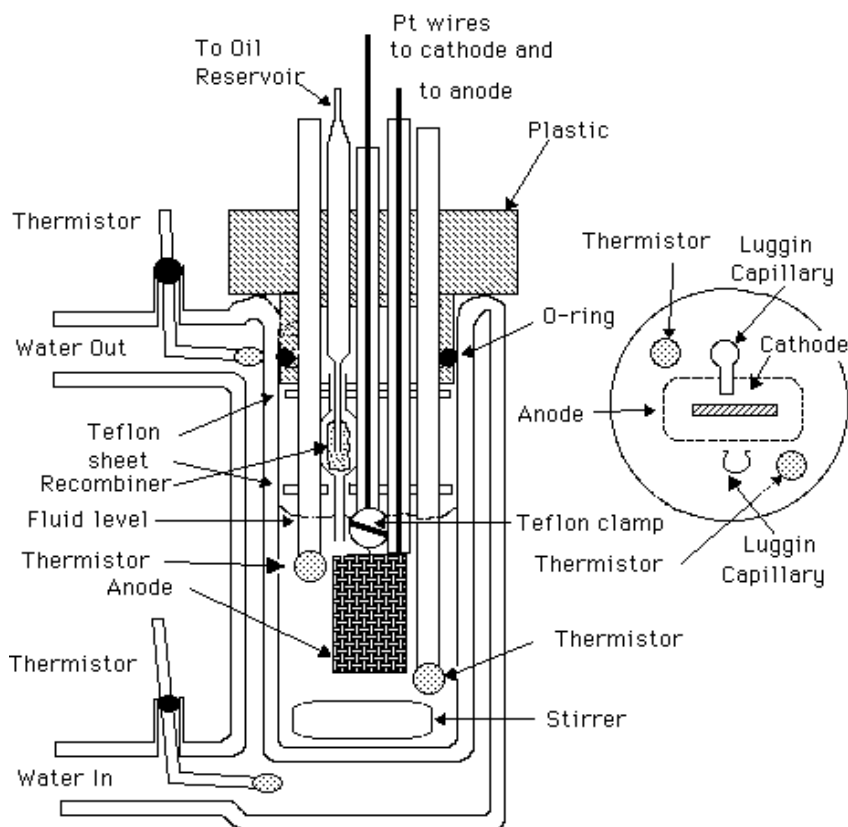

FIGURE 1. Cross-section of loading cell.

sample is large (1-2 g) and a heated Pt wire is used when vary small amounts (10-30 mg) of palladium are studied. The cell communicates with an oil reservoir which deposits oil onto a balance (±0.01 g) as unrecombined oxygen builds up within the gas space. An oil weight of 7650 ± 25 g is equivalent to 1 mol of deuterium loaded into the cathode. A small offset correction occurs because the oxygen and deuterium concentrations must reach a critical value before the recombining rate equals the production rate. For large samples, the D/Pd ratio is based on the oil displacement measurement during the run, corrected by the weight gain after the run. In such cases, the cathode was a palladium plate having dimensions of 2 cm x 1 cm

x 0.1 cm. For the plated palladium weighing approximately 10 mg, the oil system was calibrated using a clean Pt electrode before the sample was studied. All data are taken by computer using Labview.

RESULTS
Loading Efficiency

The loading efficiency is the ratio of deuterium atoms dissolved in the palladium to the number of atoms presented to the surface by the current. This ratio is determined at 5 minute intervals. Figure 2 shows the loading behavior of a Pd plate as delivered and after Aqua Regia treatment. Removal
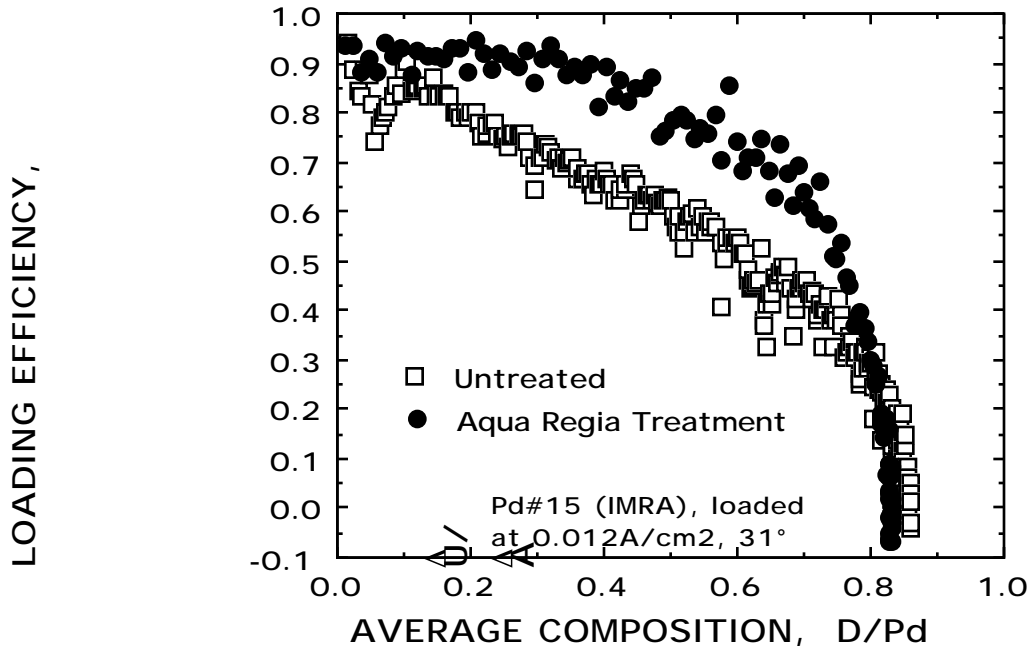
FIGURE 2. Typical loading efficiency behavior is shown for two surfaces.

of the surface barriers clearly improves the loading efficiency. Loading efficiency increases as the current is increase, at least up to 50 mA/cm^2 (Fig. 3). However, this is not the only factor. Once the steady-state composition has been reached, continued application of current causes the average composition to gradually increase. The limiting value depends on applied current (Fig.4), temperature (Fig.5), and the nature of the palladium. The nature of the palladium changes with time as impurities are deposited and as composition-induced stress is relieved.

Open-Circuit-Voltage

During loading, the OCV changes in ways that are consistent with the phase relationship (Fig. 6). If the loading rate is sufficiently small, the surface remains in equilibrium with the interior and shows a constant value within the two-phase region. As the applied current is increased, concentration gradients are introduced which modify this ideal behavior. Upon deloading, the OCV shows the reverse behavior and returns to near zero after all deuterium is lost, as shown in Fig. 7. This behavior shows that the
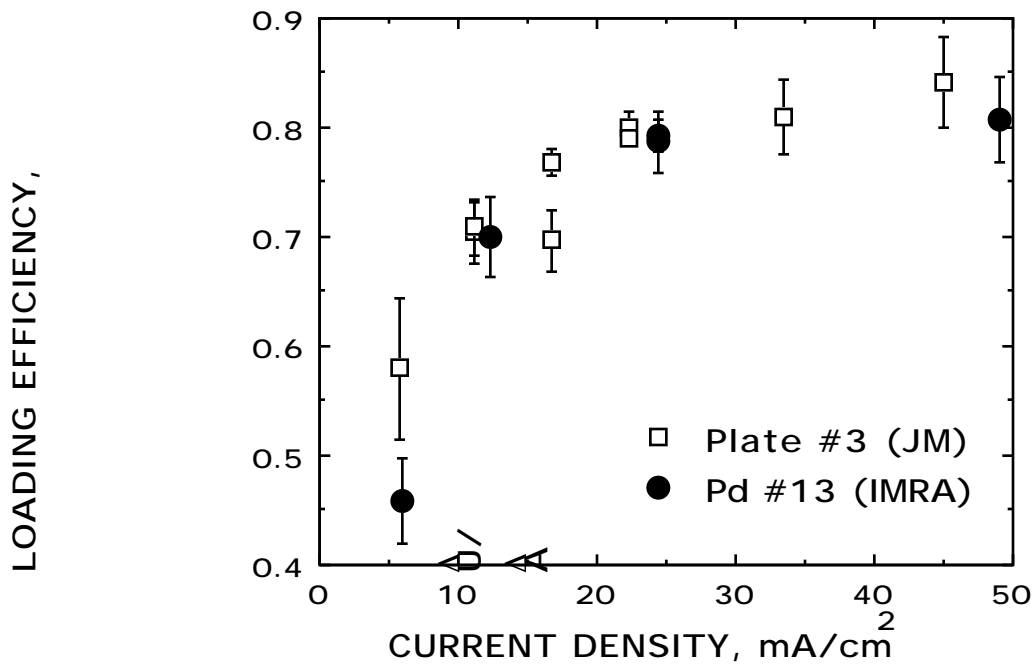
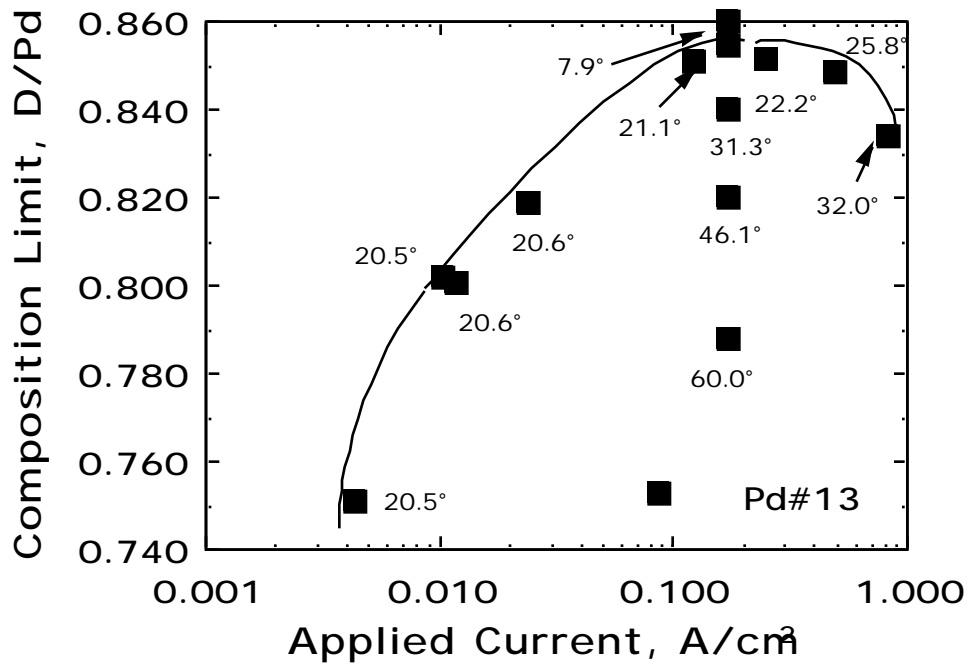**FIGURE 3**. Effect of current density on loading efficiency.

FIGURE 4. Effect of applied current on composition limit.
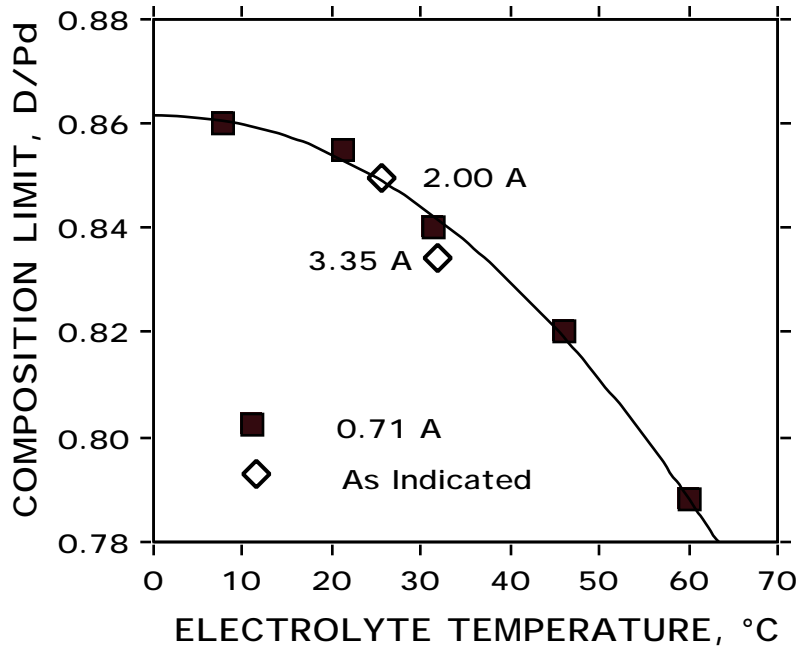The temperature of the electrolyte is shown as °C.

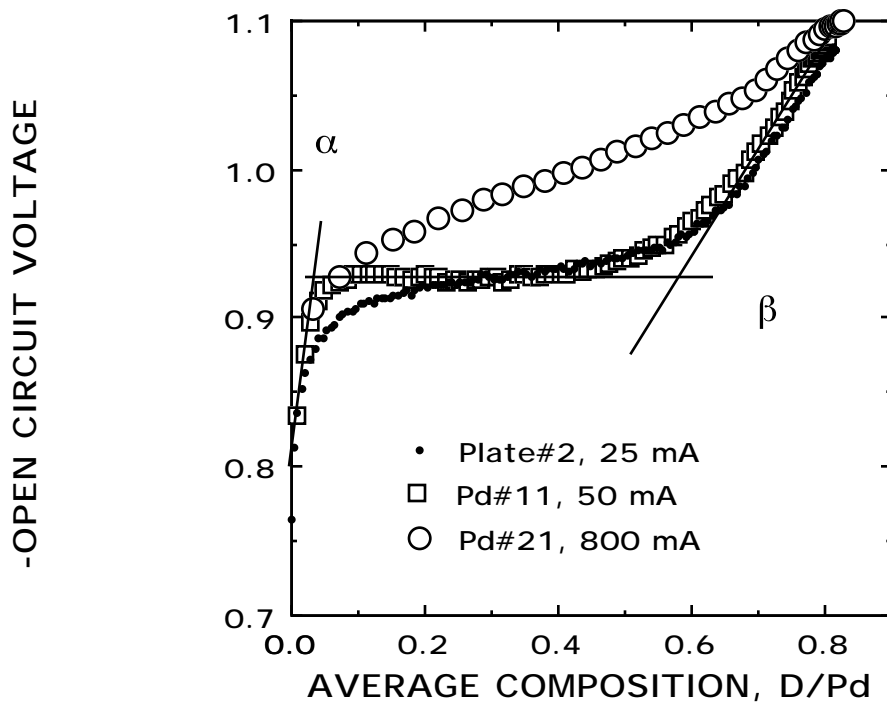**FIGURE 5.** Effect of temperature on the composition limit.

FIGURE 6. Behavior of OCV during loading at various currents.

OCV is being influenced by composition in a very direct way, hence can be used to understand composition changes at the surface.
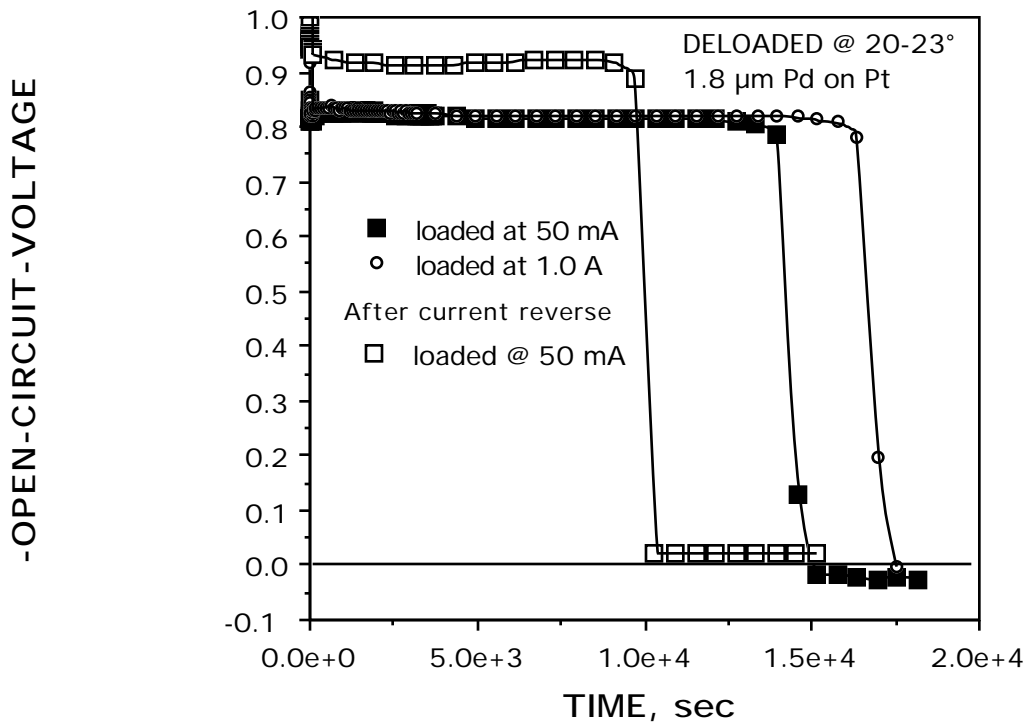
FIGURE 7. Behavior of OCV during deloading of Pd plated on Pt.

Deloading

Deloading follows a   relationship regardless of whether it occurs in air, acetone (Fig. 8) or within the cell (Fig. 9). The slope of the initial rate when
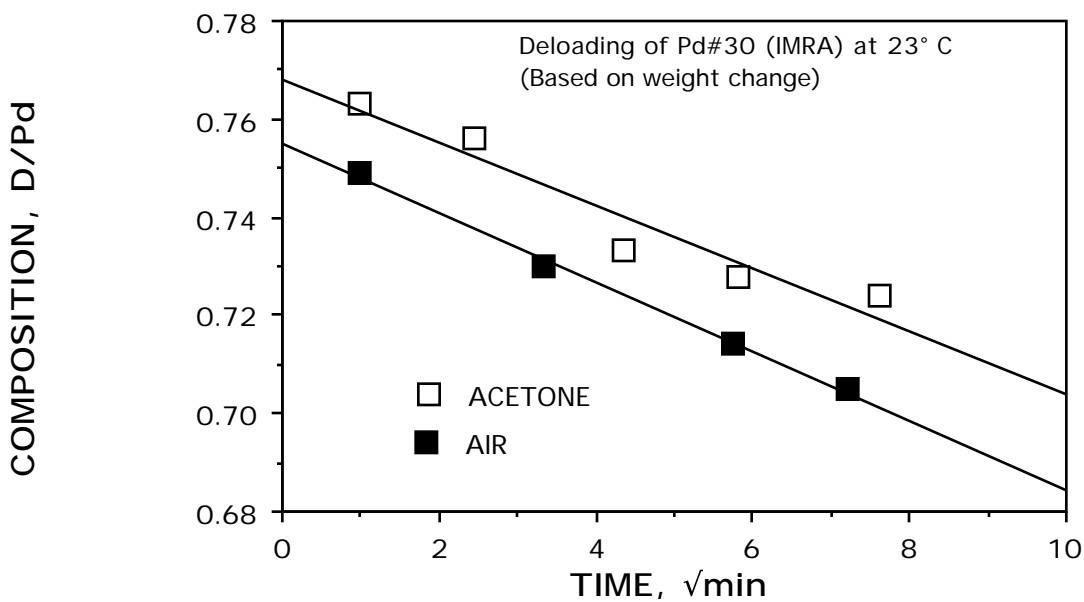

FIGURE 8. Change in average composition during deloading in air and in acetone.
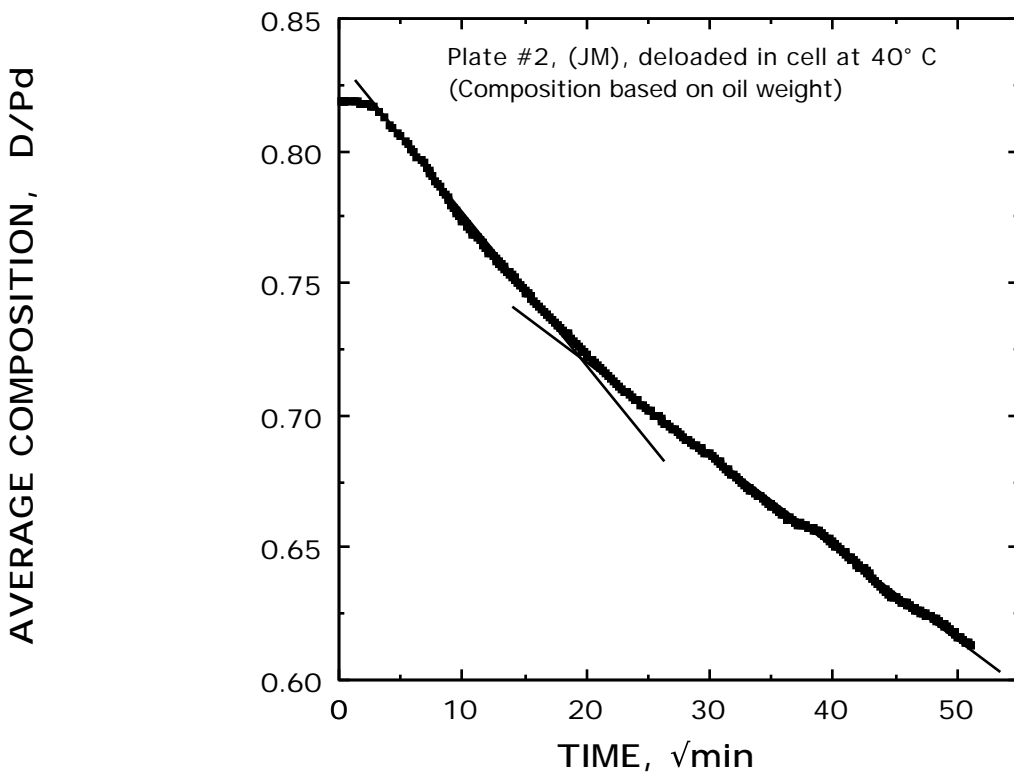
FIGURE 9. Change in average composition during deloading within the cell. The initial delay in composition change is caused by a delay in the oil system.

plotted as a function of limiting composition shows that most samples lose D at a rate which is consistent with the gas pressure within open spaces, if the concentration gradient caused by diffusion to the cracks is taken into account (Fig. 10). Occasionally, a few samples show a much smaller loss rate than expected from the limiting composition. These are the samples having the potential for making excess energy. Many samples have an initial deloading rate equal to the rate of applied deuterium produced by the current. In other words, every available D^+ diffuses into the sample and is lost as D_2 gas from the same cracks that allow loss once the current is stopped.

Surface Composition

Oil displacement was measured for pure Pt as shown in Fig. 11. The displacement of the extrapolated curve from zero is proposed to be caused by deuterium which is taken up by the platinum surface. Increased displacement as current is increased is caused by the need for a higher deuterium concentration within the gas to allow catalytic recombination to equal the increased $D_2 + O_2$ production rate. The amount of displacement produced by the platinum cathode was subtracted from the displacement produced by the electroplated sample to calculate the amount of deuterium taken up by the Pd layer The calculated D/Pd ratios for the various studies are shown in
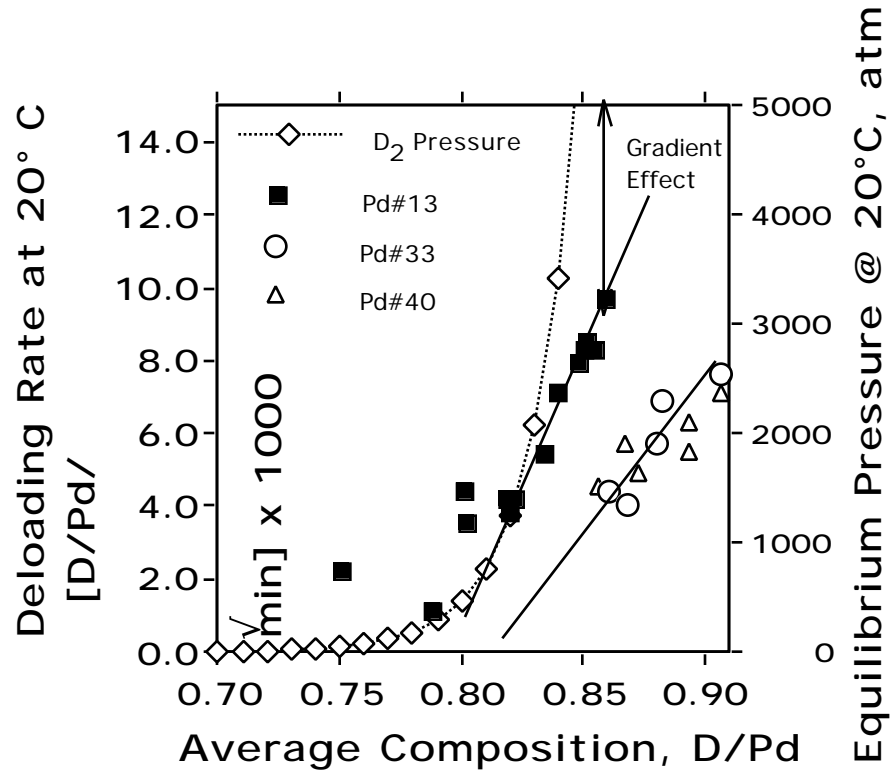
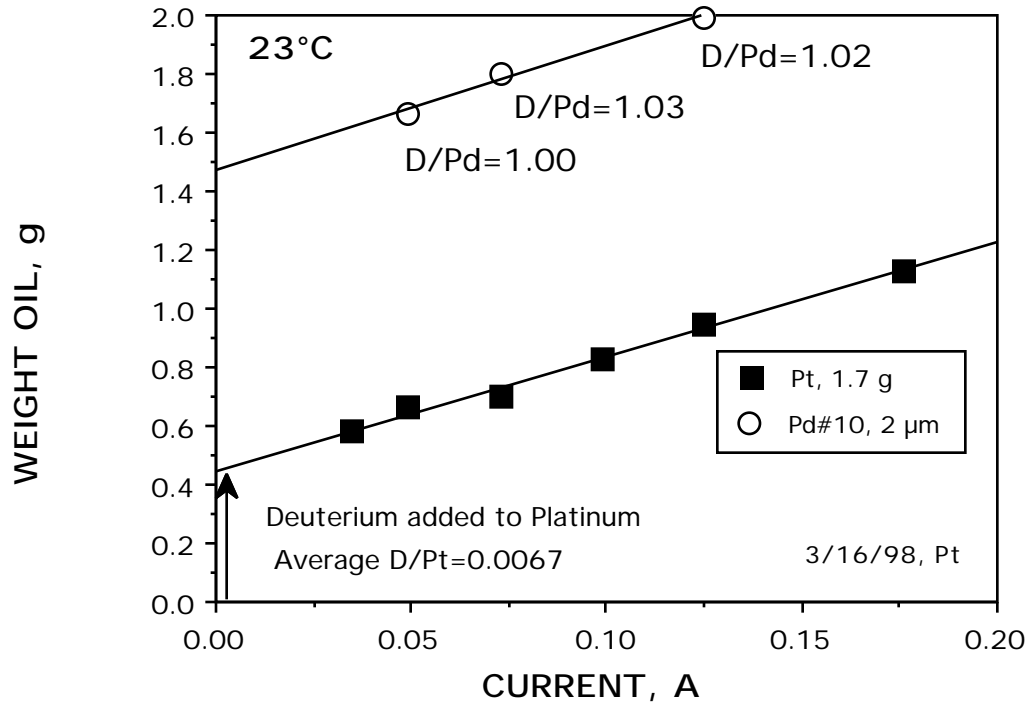**FIGURE 10.** Comparison between deloading rate and the D_2 pressure within cracks.

FIGURE 11. Oil displacement produced by pure Pt and by Pd plated on Pt. The calculated D/Pd ratios are shown for the Pd sample.

Fig. 12. This 2 µm sample was able to achieve an average composition of D/Pd = 1.5 on one occasion. Under equivalent conditions, a 50 µm foil and a 1 mm thick plate were able to reach approximately D/Pd = 0.86. The OCV of the 2 µm sample was only 0.8 V which indicates that its surface was still too low in composition to initiate a nuclear reaction. Therefore, the necessary surface composition for a nuclear reaction is in excess of 1.5, a value which is achieved only occasionally.
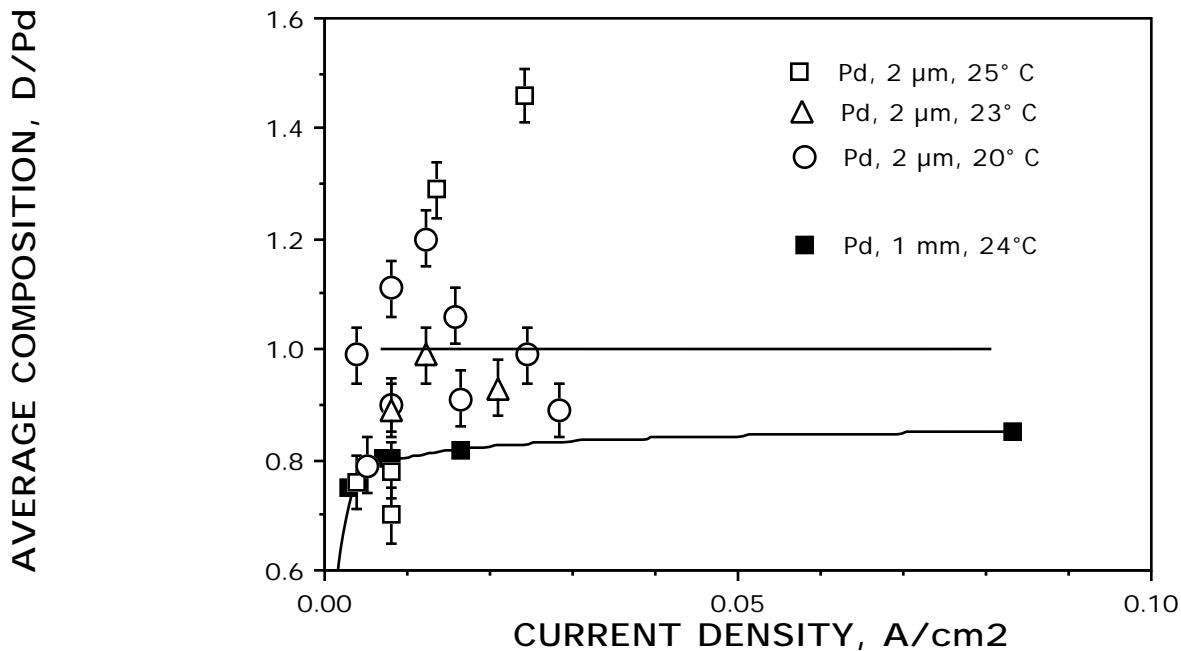

FIGURE 12. The effect of current density on the loading behavior of palladium. Cathode temperatures are shown.

CONCLUSIONS

The first problem is to find simple methods to identify palladium that is capable of achieving a high surface composition. Once such methods are available, acquiring active palladium by sorting through available palladium or by modifying production methods will become practical.

The OCV, loading efficiency, and deloading rate are powerful tools for studying the behavior of palladium. Material having a good chance of making excess energy will have (1) a OCV well above -1.06 V (relative to Pt), (2) a loading efficiency near 100% until the sample converts completely to the -phase, and (3) a deloading rate that is low compared to normal palladium at the same composition.

This and other studies indicate that essentially all deuterium presented to a clean Pd surface will be taken up by the metal, even after the composition has become constant. As the composition increases, causing an increase in the gas pressure within cracks, an increasing fraction of the applied deuterium will leave the sample as gas through surface-penetrating cracks. This process creates a concentration gradient between the surface and the bulk. Gradually, as impurities deposit on the surface of crack-free material, this gradient becomes larger until the surface acquires conditions required for energy production. Samples having a high crack concentration will not achieve this condition.

Thin layers (2 μm) of Pd plated on Pt can acquire initial, average compositions near D/Pd=1.5 at 23°C. This study will continue in order to determine the near-surface composition as a function of time, temperature, current, and layer thickness. Until the true composition and structure of the nuclear-active regions are known, there is little hope of achieving reproducible excess energy production or useful explanations.

<u>Additional Papers by the Author about CANR</u>

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