ELECTRICAL RESISTIVITY AS A FUNCTION OF DEUTERIUM CONCENTRATION IN PALLADIUM

by Gust Bambakidis, Robert J. Smith, and Dumas A. Otterson

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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ABSTRACT

The electrical resistivity of the palladium-deuterium system was measured to a deuterium- to palladium-atom ratio of 0.9 at temperatures of 273, 77, and 4.2 K. The resistivity ratio $\rho(x)/\rho(0)$ was plotted as a function of the atom ratio $x$ at 273 and 4.2 K. A modification of Mott's model for the resistivity of transition-metal alloys was used to calculate the structural resistivity. A good fit to the data at 4.2 K was obtained by assuming that the number of d-holes per palladium atom takes on the value of 0.55 to 0.60 on addition of deuterium.
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SUMMARY

The electrical resistivity of the palladium-deuterium (Pd-D) system was measured to a deuterium- to palladium-atom ratio of 0.9 at temperatures of 273, 77, and 4.2 K. The resistivity ratio $\rho(x)/\rho(0)$ was plotted as a function of the atom ratio $x$ at 273 and 4.2 K. A modification of Mott's model for the resistivity of transition-metal alloys was used to calculate the structural resistivity. A good fit to the data at 4.2 K was obtained by assuming that the number of d-holes per Pd atom takes on the value of 0.55 to 0.60 on addition of D.

INTRODUCTION

At room temperature, deuterium (or hydrogen (H)) occupies the octahedral interstitial sites of the face-centered-cubic Pd lattice (refs. 1 and 2). The octahedral sites are occupied for both $\alpha$-Pd and $\beta$-Pd, with the D/Pd and H/Pd atom ratios ranging from 0 to 0.72 (where $\alpha$-Pd and $\beta$-Pd have lattice constants of $3.86 \times 10^{-8}$ and $4.02 \times 10^{-8}$ cm, respectively). Denoting the D/Pd or H/Pd atom ratios by $x$ reveals that only $\alpha$-Pd exists for $0 < x < 0.02$. For $0.02 < x < 0.60$, $\alpha$-Pd and $\beta$-Pd coexist, and only $\beta$-Pd exists for $x > 0.60$. If $\rho(x)$ is the resistivity of PdH$_x$ (or PdD$_x$) and $\rho(0)$ the resistivity of annealed Pd, the resistivity ratio $[\rho(x)/\rho(0)]_{298K}$ and its derivative increase for increasing $x$ at $0.02 < x < 0.70$. The resistivity ratio is a maximum for $x \approx 0.76$, and the ratio along with its derivative decreases for $0.76 < x < 0.88$ (ref. 3). In the PdD$_x$ system, the available data show that the ratio $[\rho(x)/\rho(0)]_{298K}$ and its derivative increase from $x \approx 0.02$ to $x = 0.67$ with no higher values of $x$ being shown (ref. 4). For pure $\alpha$-Pd ($x < 0.02$), the derivative of the resistivity ratio $[\rho(x)/\rho(0)]_{298K}$ is larger than that for the $\alpha$-Pd + $\beta$-Pd phase for either PdH$_x$ or PdD$_x$. 
The objective of the present work is to extend the resistivity data for PdD\textsubscript{x} beyond \(x = 0.67\) to see if, in the plot of \(\rho(x)/\rho(0)\) as a function of \(x\), a maximum occurs similar to that found for PdH\textsubscript{x} at 273 K. The thermal contribution to the resistivity was minimized by obtaining data at 4.2 K, and then a modification of Mott’s model for the resistivity of transition-metal alloys (ref. 5, p. 296) was used for the analysis of the structural contribution. The resistivity results and analysis presented herein for the PdD\textsubscript{x} system should be qualitatively applicable to the PdH\textsubscript{x} system since these systems are quite similar in other resistive and crystallographic properties thus far investigated (refs. 1 to 4 and 6 to 8).

**EXPERIMENTAL PROCEDURE**

The experimental data were obtained at 4.2, 77, and 273 K. Obtaining the 4.2 K data necessitated rapidly cooling or quenching the PdD\textsubscript{x} wires from 273 to 4.2 K to prevent the migration of deuterium ions from their octahedral sites \((0, 0, 1/2; 0, 1/2, 0; 1/2, 0, 0; \ldots)\) to their tetrahedral sites \((1/4, 1/4, 1/4; 3/4, 3/4, 3/4; \ldots)\). For PdH\textsubscript{x}, an analysis of the data for neutron diffraction and that for resistivity as a function of temperature \(\rho(T)\) showed that up to 25 percent of the hydrogen may migrate to tetrahedral sites. These octahedral-tetrahedral transitions occur for PdH\textsubscript{x} at \(0.45 < x < 0.75\) if the PdH\textsubscript{x} wires are cooled without quenching (refs. 6 and 7). In the PdH\textsubscript{x} system, the octahedral-tetrahedral transitions may begin well above 100 K and continue at least to the neighborhood of 50 K (at which point a maximum in the resistivity and specific heat occurs) as the temperature is lowered from 273 to 4.2 K (ref. 6). (Whether or not additional transitions occur below these maximums is not known.) Although no neutron diffraction data are available for PdD\textsubscript{x}, the resistivity data of PdD\textsubscript{x} are almost identical to those obtained for PdH\textsubscript{x} between 273 and 4.2 K (ref. 8). However, at a given temperature, the octahedral-tetrahedral transitions take much longer in the PdD\textsubscript{x} system (up to several hours) than in the PdH\textsubscript{x} system (not more than a few seconds). From these data for PdD\textsubscript{x}, one can conclude that rapid cooling of the PdD\textsubscript{x} specimens from 298 to 4.2 K, as employed in the present experiments, was sufficient to prevent a significant amount of deuterium migration to the tetrahedral sites.

Wire specimens of 99.995 atomic percent Pd (manufacturer’s stated purity) and 0.254-millimeter diameter were annealed by joule heating in a vacuum of \(5 \times 10^{-9}\) torr at approximately 1200 K for at least 0.5 hour. Measurements of initial resistivity \(\rho(0)\) were made at 4.2, 77, 273 K by using the conventional four-probe potentiometric technique in which a phenol formaldehyde specimen holder utilized phosphorbronze spring contacts for the current and potential contacts to the specimen. Six potential contacts, spaced 1 centimeter apart between the current contacts, permitted five consecutive
measurements along the wire. This spacing allowed the detection of resistance irregularities due to preabsorption conditions and/or nonuniform absorption along the wire after deuterium absorption. The measuring currents used were 10 milliamperes in all cases.

Deuterium absorption was accomplished electrolytically with a graphite anode and a Pd-wire cathode in a 1-normal solution of deuterium sulfate ($D_2SO_4$). The current was 5 milliamperes. The anode and cathode were separated by a porous alumina crucible, as shown in figure 1. The separation was to prevent oxidizing agents that might be formed at the anode from reaching the cathode, which would permit the Pd to absorb higher concentrations of deuterium. Immediately after the absorption process, the wires were returned to the phenol formaldehyde holder for resistivity measurements at 4.2, 77, and 273 K to obtain $\rho(x)$ or the resistance $R(x)$.

For determination of the D/Pd atom ratios, each specimen was sectioned into five 1-centimeter pieces that corresponded to the positions on the phenol formaldehyde specimen holder. Then the deuterium was removed by heating each 1-centimeter section separately to 573 K in an evacuated system ($\sim 10^{-6}$ torr). Tests showed that less than 1 percent of the total gas concentration generally remained in the specimens. The specimens were weighed to three significant figures. The deuterium was expanded into and analyzed with a mass spectrometer, which is capable of 1 percent precision. The D/Pd atom ratios were then compared with their respective resistivity measurements.

![Figure 1. Container used for deuterium absorption by palladium.](image-url)
RESULTS

The results of resistivity measurements made at 4.2 and 273 K are shown in figure 2. As seen in figure 2(a), \( \frac{\rho(x)}{\rho(0)} \) at 273 K and its derivative increase with \( x \) for \( 0.02 < x < 0.65 \), and \( \frac{\rho(x)}{\rho(0)} \) at 273 K reaches a maximum in the neighborhood of \( x \approx 0.72 \) and then decreases as \( x \rightarrow 0.90 \). At 4.2 K, the maximum in \( \frac{\rho(x)}{\rho(0)} \) at 4.2 K appears to be near a value of 0.68 for the D/Pd atom ratio.

![Graph showing resistivity ratio as a function of deuterium concentration in palladium.](image-url)

Figure 2. - Resistivity ratio as function of deuterium concentration in palladium.
Some of the scatter in the data may be attributed to two sources: (1) some hydrogen contamination, which varied between 3 and 8 percent of the total gas concentration, and (2) some evolution of the absorbed deuterium, for $x > 0.7$, between the resistivity measurements and mass spectrographic analysis. The time period for which evolution could take place was less than 20 minutes. (There is no evidence that evolution occurred at or below 77 K.) The possible gas loss for this time period is approximately a D/Pd atom ratio of 0.025, which is equivalent to a 2-percent change in $\rho(x)/\rho(0)$ at 273 K. The data shown in figure 2 include both the deuterium and the hydrogen impurity in $x$ (i.e., $(D+H)/Pd$).

**DISCUSSION**

In figure 2(a), an experimental curve was drawn through the data points, whereas the curves in figure 2(b) were calculated for the values 0.36 and 0.55 for the number of holes $n_h$ per Pd atom in the d-band. (The results for 77 K are not given because some octahedral-tetrahedral transitions occur during measurement.) The difference in the data at the two temperatures shown is attributed to the thermal contribution to the resistivity. At 4.2 K, both the thermal resistivity and that arising from lattice defects are expected to be small, so that their variation with deuterium concentration can be neglected. If $x$ denotes the D/Pd atom ratio, the resistivity ratio $\rho(x)/\rho(0)$ at this temperature can therefore be written

$$\frac{\rho(x)}{\rho(0)} = 1 + \frac{\rho_D(x)}{\rho(0)}$$

where $\rho_D(x)$ is the contribution from disorder scattering by the deuterium randomly distributed among the octahedral sites in the host face-centered-cubic Pd lattice. This quantity is also the resistance ratio because the change in sample dimensions due to lattice expansion on absorption of D is negligible here. The average value of $\rho(0)$ for the samples used was determined to be 0.105 microhm-centimeter.

Magnetic susceptibility (ref. 5, p. 189) and electromigration (ref. 9) studies on the Pd-H system indicate that H exists in the Pd lattice as strongly screened positive ions, and presumably the same situation exists for D. The electronic structure of pure Pd was described by use of the Mott model (ref. 5, p. 189), in which the band structure is assumed to consist of a broad free-electron-like s-band overlapping with a narrow d-band, so that the Fermi level occurs near the top of the d-band. Thus, there are electrons in the s-band and an equal number of holes in the d-band. The assumption is made that initially the D atoms contribute their electrons to fill the holes in the d-band.
In a covalent bond picture, the electron is thought of as being shared with neighboring Pd atoms, which results in a strong screening of the D ion. The D ions, therefore, scatter a conduction electron only weakly, giving rise to a small initial slope (the change in slope on appearance of the β-phase, observed at higher temperatures, is greatly diminished at 4.2 K, which suggests that its origin is thermal rather than structural). As the holes in the d-band are filled, the electrons are instead contributed to the s-band, whose states are delocalized and, hence, do not screen as strongly. This lack of screening results in the sharp rise in resistance. Thus, the resistance is viewed as arising from scattering by three types of local structure, denoted by I, II, and III in the potential energy diagram of figure 3. Each of the three types is distributed randomly throughout the system. The screened coulomb potentials about the D ions have screening lengths $a_d$ and $a_s$, with $a_d < a_s$.

If the current carried by the holes is neglected, the use of the Boltzmann equation and the assumption of an isotropic relaxation time $\tau$ lead to the usual expression

$$\rho = \frac{m_e^*}{N_e e^{2\tau}}$$

where $m_e^*$ is the effective mass and $N_e$ the number of conduction electrons per unit volume. The inverse of the relaxation time can be written
However, the contribution from s-d scattering, proportional to the density of states at the Fermi level, goes to zero as the d-band fills. We therefore feel that s-d scattering cannot play a significant role in the sharp rise in $\rho(x)/\rho(0)$ with $x$ and neglect it entirely.

When $1/\tau_{s-s}$ is evaluated in Born approximation (see appendix), the result for $\rho_D(x)$ is

$$
\rho_D(x) = \frac{2}{3\pi} \frac{m_e^*e^2}{\hbar^3} \frac{\Omega_0(x)}{[n_e(x)]^2} \left[ f\left(4k_f^2a_d^2\right)x_d\left(1 - x_d\right)
+ f\left(4k_f^2a_s^2\right)x_s\left(1 - x_s\right)
+ 2g(k_f,a_d,a_s)x_d^s x_s
\right]
$$

The quantity $\Omega_0(x)$ is the volume per Pd atom which is weakly dependent on $x$ because of lattice expansion. Because the exact variation of lattice parameter with concentration is not important here, a simple linear expansion of the lattice parameter from $3.88 \times 10^{-8}$ centimeter at $x = 0$, to $4.02 \times 10^{-8}$ centimeter at $x = 0.60$, is assumed. The quantity $n_e(x)$ is the number of electrons per Pd atom in the s-band. The Fermi radius $k_f$ is given by

$$
k_f = \left[ \frac{3\pi^2n_e(x)}{\Omega_0(x)} \right]^{1/3}
$$

while $f$ and $g$ are defined by

$$
f\left(4k_f^2a_s^2\right) = \ln \left(1 + 4k_f^2a_s^2\right) - \frac{4k_f^2a_s^2}{1 + 4k_f^2a_s^2}
$$

and

$$
g(k_f,a_d,a_s) = \frac{a_s^2 \ln \left(1 + 4k_f^2a_d^2\right) - a_d^2 \ln \left(1 + 4k_f^2a_s^2\right)}{a_s^2 - a_d^2}
$$
The coefficients $x_d$ and $x_s$ denote the number of D atoms per Pd atom contributing an electron to the d-band and s-band, respectively. Both depend implicitly on the total concentration $x$.

Expression (4) cannot be expected to be valid in the upper range of concentration (at and above the maximum in $[\rho(x)/\rho(0)]_{4,2K}$) because the development of long-range order near $x = 1$, where the system takes on the sodium chloride structure, was not taken into account. The expression, therefore, overestimates the resistance in this range. If at $x = 1$, type I and II structures are regarded as constituting independent superlattices, then the resistance for $x \leq 1$ will arise from scattering by type II and III structures only. Proceeding as before in the evaluation of $1/\tau_{S-S}$ results in the high-charge expression (see appendix)

$$\rho_D(x) = \frac{2}{3\pi} \left(\frac{m_e^*}{h^3}\right) \frac{e^2}{\Omega_o(x)} \left(4k_F^2a_s^2\right) \frac{(x - n_h)(1 - x)}{1 - n_h} \quad (5)$$

where $x \geq n_h$. The parameter $n_h$ is the number of holes per Pd atom in the d-band at the initiation of charging; hence, it is also the number of octahedral sites per Pd atom available to the strongly screened D ions. The assumption of independent superlattices means that equation (5) underestimates the resistance in the range $x \geq n_h$.

The terms $x_d$, $x_s$, and $n_e$ are expected to have the qualitative behavior shown in figure 4. Note that $x_d + x_s = x$ and that $n_e(x) = n_e(0) + x_s$. The concentration $x_f$ is that at which the d-band fills, and experiments by others (ref. 5, p. 316) on palladium-noble-metal alloys indicate $x_f$ to be in the region of 0.55 to 0.60. On the basis of the

![Figure 4. Expected total-concentration dependence of quantities $x_d$, $x_s$, and $n_e$ entering into equations (4) and (5).](image)
de Haas - van Alphen measurements of Vuillemin (ref. 10) on pure Pd, $n_e(0) = 0.36$. If this value is also used for the number of holes, the upper curve in figure 2(b) is obtained. The dashed line indicates the continuous curve that would result from a more exact treatment of the onset of long-range order. The effective mass used, which was assumed independent of composition, was an average band mass of 0.49 $m_e$ obtained from $k_f$ and the Fermi level $E_f$ for pure Pd. The quantity $E_f$ was taken as 0.462 rydberg by using Segall's (ref. 11) band-structure calculations for copper and the similarity in the band structures of Pd and Cu (ref. 10). The quantity $x_f$ was taken as 0.60, and $a_d$ and $a_s$ were determined by fitting equations (4) and (5) to the initial slope and final (extrapolated) slope, respectively. The fit over the entire concentration range is certainly not quantitative, but if the number of holes in the d-band is assumed to increase from 0.36 on charging, a much better fit is obtained, as shown in figure 2(b) by the curve for $n_h = 0.55$. Values for $n_h$ in the range 0.55 to 0.60 are consistent with magnetic susceptibility data obtained by others on Pd-H (ref. 5, p. 316). The values for the screening lengths are $a_d = 0.26$ atomic units and $a_s = 1.11$ atomic units (at $x = 1$). This value for $a_s$ is somewhat larger than the Thomas-Fermi value of 0.74 atomic units.

The authors are aware that the assumption of a spherical s-electron Fermi surface neglects the appreciable anisotropy, which extends to about 30 percent (ref. 10). However, the Mott model, with the interpretation of the de Haas - van Alphen work that $n_e(0) = n_h(0) = 0.36$, appears to work well in explaining the transport properties of the Pd - noble-metal alloys (ref. 12). The apparent failure of the rigid-band model for Pd-D indicated herein suggests that further work on this system would be of interest. In particular, a band-structure calculation could be done for the ordered structure $x = 1$ to obtain $n_e(1)$. The rigid-band model predicts one s-electron per Pd atom at D/Pd or at H/Pd = 1, whereas the present analysis would indicate 0.81 electron per atom. In conjunction, a de Haas - van Alphen experiment could be conducted, which would be dependent, however, on overcoming present experimental difficulty in consistently obtaining high D/Pd ratios.

CONCLUSIONS

The direct-current electrical resistivity of the palladium-deuterium (Pd-D) system was measured to a deuterium- to palladium-atom ratio of 0.9. Analysis of the resistivity as a function of atom ratio at 4.2 K indicated that on addition of deuterium, the number of holes in the d-band (per Pd atom) takes on the value of 0.55 to 0.60, compared with the value of 0.36 for pure Pd, which is based on recent experimental and theoretical work by
others. This result suggests that the rigid-band model is not valid for the Pd-D interstitial solid solution, in contrast to the Pd-noble-metal alloy systems.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 16, 1968,
129-03-15-01-22.
APPENDIX - EVALUATION OF INVERSE OF RELAXATION TIME

The inverse of the relaxation time $\tau_{s-s}$ for scattering of an s-electron at the Fermi surface back into an s-state by a single scatterer is (ref. 5, p. 247)

$$\frac{1}{\tau_{s-s}} = \frac{\Omega}{4\pi^2 \hbar} \int_{s\text{-surface}} dS(1 - \cos \theta)|v_{k'k}|^2 \frac{1}{|\nabla E_f|}$$

(A1)

$$= \frac{\Omega}{2\pi \hbar} k_f^2 (\frac{dE}{dk})_{k=k_f}^{-1} \int_0^\pi d\theta (1 - \cos \theta) \sin \theta |v_{k'k}|^2$$

The integral is taken over all scattering angles $\theta$ between the initial state $\underline{k}$ and final state $\underline{k'}$. The matrix element $v_{k'k}$ is

$$\int_{\Omega} d\underline{r} \, \varphi^*_k(r) v(r) \psi_k(r)$$

where $\psi_k$ is the initial state in the presence of the scattering potential $v(r)$, and $\varphi_{k'}$ is the final state in the absence of $v$. The s-band wave functions are assumed to be plane waves. In the Born approximation, $\psi_k$ is taken equal to $\varphi_k$ in evaluating $v_{k'k}$. Hence,

$$v_{k'k} = \frac{1}{\Omega} \int_{\Omega} d\underline{r} \, e^{-i(k' - k) \cdot \underline{r}} v(r)$$

(A2)

Since a perfectly periodic potential has no resistance, the contribution from each type of local structure of figure 3 is considered to arise from the difference between the actual potential and the average potential there. The average potential is

$$v_{av} = \left[v_{pd} + v_D(a_d)\right]x_d + \left[v_{pd} + v_D(a_s)\right]x_s + v_{pd}(1 - x_d - x_s)$$

Hence, the scattering potentials are
Type I:

\[ v(r) = v - v_{av} \]

\[ = \left[ v_{Pd} + v_D(a_d) \right] - \left[ v_{Pd} + x_d v_D(a_d) + x_s v_D(a_s) \right] \]

\[ = (1 - x_d)v_D(a_d) - x_s v_D(a_s) \]

Type II:

\[ v(r) = (1 - x_s)v_D(a_s) - x_d v_D(a_d) \]

Type III:

\[ v(r) = -x_d v_D(a_d) - x_s v_D(a_s) \]

The total contribution to \( 1/\tau_{S-S} \) from each type of scatterer consists of a term of the form of equation (A1) with the appropriate scattering potential, weighted by the number of each type. Denoting matrix elements by \( \langle \cdot \rangle \) and integration over the scattering angle by \( \theta \) gives

\[
\frac{1}{\tau_{S-S}} = \frac{\Omega N_{Pd}}{2\pi h} k_f^2 \left( \frac{dE}{dk} \right)_{k=k_f}^{-1} \left\{ \left| \langle v_D(a_d) \rangle \right|^2 x_d (1 - x_d) + \left| \langle v_D(a_s) \rangle \right|^2 x_s (1 - x_s) \right. \\
+ 2 \text{Re} \left[ \langle v_D(a_d) \rangle \langle v_D(a_s) \rangle^* \right] x_d x_s \right\} \tag{A3}
\]

where \( N_{Pd} \) is the number of Pd atoms in the system. The appropriate integrals can be evaluated in a straightforward manner, the result being equation (4) of the text.

In the high-charge region, the resistance is assumed to arise mostly from the presence of vacancies in a superlattice of type II structures. The scattering arises from fluctuations in an average potential:

\[ v_{av} = v_{Pd} + \frac{x_s}{1 - n_h} v_D(a_s) \]

The scattering potentials are now
Type II:

\[ v(r) = \left(1 - \frac{x_s}{1 - n_h}\right) v_D(a_s) \]

Type III:

\[ v(r) = -\frac{x_s}{1 - n_h} v_D(a_s) \]

There are \( x_s \) type II scatterers and \( 1 - x \) type III scatterers per Pd atom. Therefore,

\[
\frac{1}{\tau_{s-s}} = \frac{\Omega N_{Pd}}{2\pi \hbar} k_f^2 \left( \frac{dE}{dk} \right)^{-1}_{k=k_f} \left[ \left| \langle v_D(a_s) \rangle \right|^2 \left( 1 - \frac{x_s}{1 - n_h} \right)^2 x_s \right. \\
\left. + \left| \langle v_D(a_s) \rangle \right|^2 \left( \frac{x_s}{1 - n_h} \right)^2 (1 - x) \right] \tag{A4}
\]

Now, \( x_d + x_s = x \), and from figure 4, \( x_d = n_h \) for \( x \gg n_h \). Thus, using \( x_s = x - n_h \) results in

\[
\frac{1}{\tau_{s-s}} = \frac{\Omega N_{Pd}}{2\pi \hbar} k_f^2 \left( \frac{dE}{dk} \right)^{-1}_{k=k_f} \left[ \left| \langle v_D(a_s) \rangle \right|^2 \frac{(x - n_h)}{1 - n_h} \frac{(1 - x)}{x \gg n_h} \right] \tag{A5}
\]

Substituting the explicit expression for \( \left| \langle v_D(a_s) \rangle \right|^2 \) leads to equation (5) of the text.
REFERENCES


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