The effect of hydrogen stoichiometry on palladium strain and resistivity

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A B S T R A C T

The strain and the electrical resistivity of a Pd sample stressed by a constant tension have been investigated through a series of hydrogenation cycles in a continuous H stoichiometry $[0 \leqslant x \leqslant 0.8]$ range. The isotropic lattice expansion for both "as drawn" and "annealed" Pd sample reveals a strain of only 1% from pure Pd to $PdH_{0.8}$ in disagreement with literature data available; the measured effect is minimum at $x = 0.13$ ($\alpha + \beta$ phase) and then from $x = 0.6$ (β phase) it has an exponential increase. The contribution of the mechanical tensile stress on the total relative elongation of the wire is also investigated. An increase of the Pd sample tensile strain after each hydrogenation cycle is reported for "as drawn" samples, while for "annealed" samples the reverse behaviour is observed. Moreover, annealed samples show considerably higher value of tensile strain compared to "as drawn". The variation of mechanical strain versus H content, for both "annealed" and "as drawn", has a maximum at $x = 0.52$. Strain variation and resistivity variation versus H content exhibit similar behaviour.

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1. Introduction

It's well known that H dissolves in Pd occupying interstitial sites in the host lattice, producing an expansion of the lattice and vacancies which affect the mechanical and electrical characteristics of the Pd sample [1–4]. Recently [5], for a sample stressed by a constant tension, has been reported that a series of hydrogenation and dehydrogenation cycles ($\alpha \rightarrow \beta \rightarrow \alpha$ phase transitions) cause a large increase of the residual Pd relative elongation $\Delta l/l$ of 8.8% while sample only stressed shows $\Delta l/l$ of 0.3%. The H cycles cause up to 15% [5] Pd residual resistivity increase. It's obvious that mechanical and electrical variation of Pd are attributable to the H diffusion defects production and the stress applied to the sample [5]. Oppositely, in absence of tension applied, the electrical resistance of the Pd sample decreases after H cycles [6]. In which way the absorption of H in Pd affects the expansion of the lattice at room temperature is very divergent in the literature. Most of the available data report results through XRD and dimensions measurement showing an overall expansions of the lattice parameter of about 3.5–4% (10–12% in volume) that corresponds to H contents in the β phase [2,10,16–20] range. Another work shows an unexpected excess volume of 18% in β phase, supposedly due to voids and defects creation [21]. Recently some authors show lower values of lattice [22] expansion and a relative elongation $\Delta l/l$ of about 1.5% in β phase at 313 K [23]. Larger Pd clusters ($\approx$6 nm) show much higher lattice expansion values in comparison to those of the small clusters ($\approx$3 nm) at the same H stoichiometry [24,25]. This difference in behaviour is attributed to the different clusters structure.

Data for Young modulus and breaking strain is examined for Pd at room temperature as a function of the H equilibrium solubility, where Pd shows a ductility more pronounced in α phase, followed by decreasing values of about 20% in $\alpha + \beta$ phase [7]. On the other hand, the mechanical hardness, as a function of H contents introduced by electrolysis, for a cold-worked Pd sample, is found to increase in α phase and then almost comes back to its initial value in the β phase [7]. Anyway, a limited literature is available on the applied tensile stress effect for Pd during H loading [8,9]. The objective of this study is to determine the role of the hydrogenation process on Pd properties change (strain and resistivity) by separating the isotropic expansion due to H insertion from the strain caused by the applied tensile stress. The variation of these properties will be investigated for both "as-drawn" and "annealed" Pd samples.

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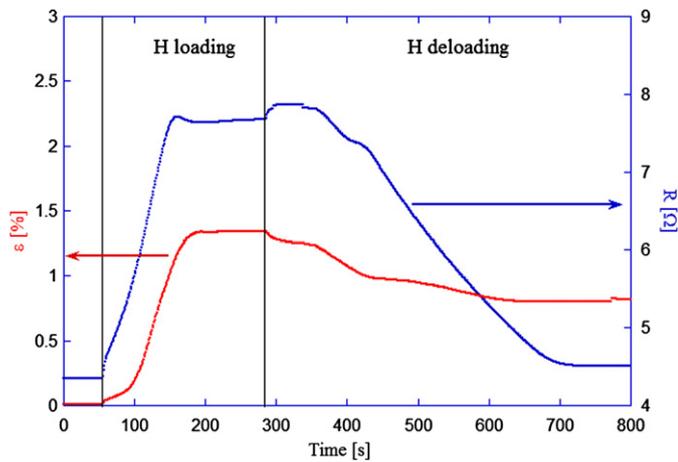
Fig. 1. Relative elongation (red) and Resistance (blue) of the Pd wire versus time for the first hydrogenation (cathodic 10 V)/dehydrogenation (anodic 3 V) cycle. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

The set-up and the electrolytic cell used in this work have been described elsewhere [5]. The results presented in this Letter are obtained with same batch Pd. In this Letter, the term "annealing" refers to the following procedure: the Pd wire is heated at room atmosphere by Joule effect, applying a current of 650 mA. A linear increase of the current from 0 to 650 mA is applied to the wire for 5 minutes, and then maintained at this level for 5 minutes followed by the decrease from 650 to 0 mA again in 5 minutes.

2. Results

The objective of these experiments is to study, in a continuous range of stoichiometry, the effect of H content on the lattice expansion and on the electrical resistivity variation of Pd samples. The variation of these physical properties is investigated for several H loading cycles on the same Pd samples.

Hydrogenation of the Pd wires has been performed by means of cycles consisting in H loading of Pd cathode and subsequent H deloading from it. The experiments [5] were performed with the Pd wire stressed by a constant tension $T \approx 0.49$ N, equivalent to an initial pressure of about 260 MPa. Seven cycles of hydrogenation/dehydrogenation were performed before the rupture of the wire occurred.

Fig. 1, shows the Pd wire resistance measurement and the length variation versus time during the first hydrogenation/dehydrogenation cycle. The H loading inside the Pd wire is obtained by potentiostatic electrolysis. As expected, the introduction of H into the lattice causes the Pd resistance increase [1], and the expansion of the Pd lattice [2]. On the contrary, applying a reverse difference of potential we obtain the dehydrogenation of the Pd wire, and the subsequent decrease of both Pd wire resistance and length.

As shown in a previous study [5], at the end of each cycle an increase of the residual elongation of the wire (up to a strain of 8.8% after seven cycles) and of its resistivity (up to 15% after seven cycles) is observed, due to a progressive internal stress of the stretched Pd sample. Moreover this residual strain ε is a linear function of the resistivity ρ [5], according to the Matthiessen's rule [11,12]:

$$\frac{\Delta \rho}{\rho_{0,0}} = \frac{\rho_{0,n} - \rho_{0,0}}{\rho_{0,0}} = K_H \cdot \varepsilon, \qquad (1)$$

where $\rho_{0,0}$ is the initial resistivity of the sample, and $\rho_{0,n}$ is the resistivity of the wire at the end of the nth hydrogenation/dehydrogenation cycle, and K_H are constants. Experimental
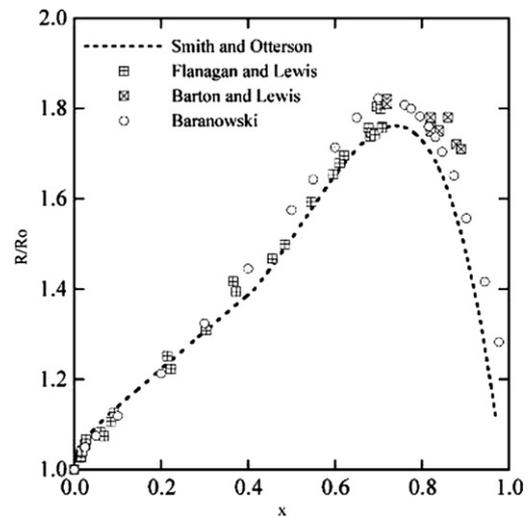
Fig. 2. Relationship between relative resistance and loading factor at 298 K obtained for four different sources [8].
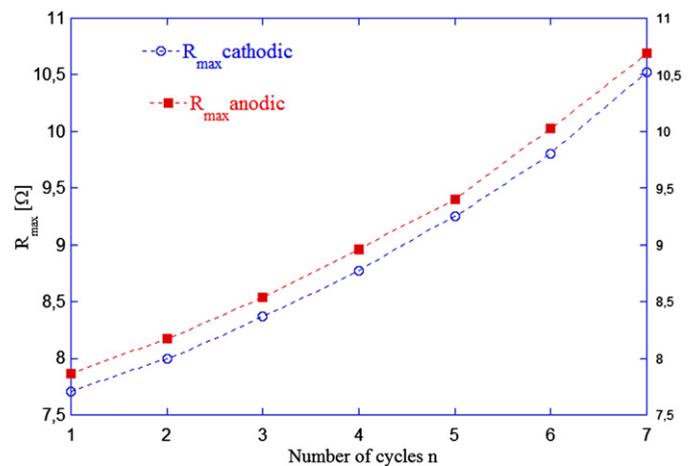
Fig. 3. Resistance value at PdH$_{0.73}$ for loading (cathodic 10 V) and deloading (anodic 3 V) versus number of cycles.

data shows that the constant K_H is at least one order of magnitude higher than the same constant obtained without the H cycle dynamics [5]. The relationship between the electrical resistance of a Pd sample loaded with H and the H concentration is always expressed in terms of the relative electrical resistance, R_x/R_o, where R_x is the resistance of Pd loaded at x stoichiometry (PdH$_x$), and R_o is the resistance of Pd when it is H free, as shown in Fig. 2 [8].

AC electrical resistance measurement is an indirect method to determinate the H content dissolved in Pd lattice. The advantage of this method is that the resistance reflects the overall bulk properties of the sample affected by the H adsorption. A polynomial function [6] can be used to fit the resistance ratio R_x/R_o as a function of $[0 \leqslant x \leqslant 0.8]$, based on the experimental data in Refs. [13,14], then it is possible to determine the average stoichiometry x from our relative resistance measurements with an error $\Delta x = 0.01$. Fig. 1 shows, that in the region of H loading there is a maximum of resistance at a stoichiometry of about $x = 0.73$. At the same stoichiometry, in the region of deloading, we observe a maximum value of the resistance higher than the one obtained during the H loading. This behaviour is supposed to be due to a difference of distribution of H concentration when it is adsorbed or desorbed by the host lattice.

The values of maximum resistance increase after each H cycle with the same trend for both loading and deloading (circles and
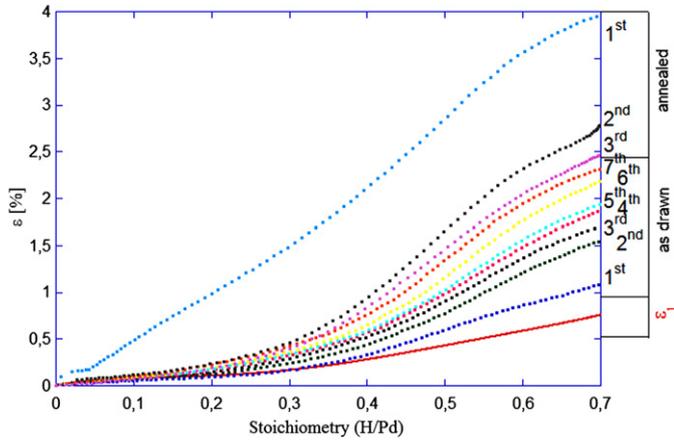
Fig. 4. Strain due to the lattice expansion (red line, fit of Fig. 5), and total relative elongation for "as drawn" and "annealed" samples as a function of H stoichiometry for a series of hydrogenation loadings (cathodic 10 V) on the same Pd sample (maximum error on ε: ±0.02). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this Letter.)
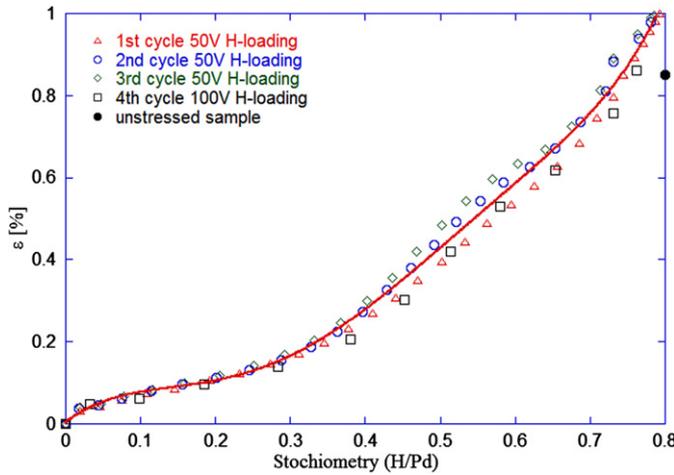
Fig. 5. Strain due to the lattice expansion and fit (red line) for three H loadings (cathodic 50 V) and one H loading (cathodic 100 V) on the same Pd sample as a function of the stoichiometry (maximum error on ε_l: ±0.02). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this Letter.)

squares of Fig. 3), confirming the progressive increase of the internal stress, as reported in the previous studies [5]. During each hydrogenation cycle, the H adsorption into the Pd host lattice not only increases its resistance, but at the same time it also causes the expansion of the lattice. The extent of the lattice expansion can be quantified by the relative elongation $\Delta l/l$, defined as the ratio between the length variation of the sample to the length of the sample when free from H. Since Pd lattice is fcc, an isotropic expansion is assumed. In the loading region, the length of the wire increases as soon as H is inserted into Pd lattice (PdH resistance increases), and reaches its maximum value when the concentration of H inside the host lattice is constant (PdH resistance is stable). During the H deloading the reverse behaviour is observed. This means that the observed deformation of Pd lattice is strictly connected to the H dynamics during the H loading and deloading. To our knowledge, the elongation of the sample in relation to its H content has not been widely investigated in this range of stoichiometry at room temperature. Moreover, there is a poor literature related to the influence of a tensile stress applied to a pure Pd sample on the wire relative elongation when it is loaded with H.

We focus our study in a continuous range of stoichiometry between $x = 0$ (α phase) to about $x = 0.8$ (β phase), analysing the role of the applied tensile stress on the observed relative elongation.

Instantaneous measurement of the relative resistance R_x/R_o, and the sample length variation Δl allows us to investigate the relationship between the deformation of the Pd wire, $\varepsilon = \Delta l/l$, and the H concentration inside the host lattice (Fig. 4). Data shows that the elongation of PdH$_x$ sample is an increasing monotone function of the H content. An increase of the strain referred to the same H concentration is observed cycle after cycle, confirming a progressive increase of lattice internal stress. In fact, an increase of the residual strain is observed, as well as a decrease of the cross section of the wire [5] at the end of each cycle. The strain ε, during the hydrogenation of a stretched sample, is produced by the combined effect of the lattice expansion $\varepsilon_l = (\Delta l/l)_l = \Delta a/a$ due to H insertion, where a represents the lattice constant, and $\varepsilon_s = (\Delta l/l)_s$ the strain resulting from a tensile mechanical stress. Since Pd has an fcc structure, the first one has an isotropic effect on the expansion of the Pd lattice in the three axis directions x, y, z, considering the length of the wire in the z axis:

$$\left(\frac{\Delta l_x}{l_x}\right)_l = \left(\frac{\Delta l_y}{l_y}\right)_l = \left(\frac{\Delta l_z}{l_z}\right)_l$$
$$\Rightarrow \quad \frac{\Delta S}{S} = \left(\frac{\Delta l_x}{l_x}\right)_l + \left(\frac{\Delta l_y}{l_y}\right)_l = 2 \cdot \left(\frac{\Delta l_z}{l_z}\right)_l = 2 \cdot \varepsilon_l. \quad (2)$$

On the contrary, the mechanical tensile stress does not affect the lattice constant, but displaces the lattice planes causing an irreversible increase of the length and a decrease of the wire's cross section according to the Poisson ratio ν [5]:

$$\left(\frac{\Delta l_x}{l_x}\right)_s = \left(\frac{\Delta l_y}{l_y}\right)_s = -\nu \cdot \left(\frac{\Delta l_z}{l_z}\right)_s$$
$$\Rightarrow \quad \frac{\Delta S}{S} = \left(\frac{\Delta l_x}{l_x}\right)_s + \left(\frac{\Delta l_y}{l_y}\right)_s = -2 \cdot \nu \cdot \left(\frac{\Delta l_z}{l_z}\right)_s$$
$$= -2 \cdot \nu \cdot \varepsilon_s, \quad (3)$$

where for Pd $\nu = 0.39$ [15].

The values of total strain ε and the expansion of the lattice ε_l allow us to calculate the effect of the strain ε_s produced by the mechanical tensile stress:

$$\varepsilon_s(x) = \frac{\varepsilon(x) - \varepsilon_l(x)}{1 + \varepsilon_l(x)}. \quad (4)$$

In order to investigate the extent of the Pd lattice expansion in relation to its H content, and consequently to separate the effect of ε_s and ε_l, we performed an experiment with 10 V, 50 V and 100 V potentiostatic electrolysis applying no mechanical tensile stress and measuring the length of Pd wire only before ($x = 0$) and at the end ($x = 0.8$) of the hydrogenation cycle. In this condition, the contribution of the strain is only due to the lattice expansion ε_l, we observed an elongation $\varepsilon_l = 0.85\%$.

Moreover we performed another experiment on a Pd stretched wire ($T = 0.49$ N), obtaining the same maximum value of stoichiometry ($x = 0.8$) by increasing the electrolysis cathodic voltage to 50 V and 100 V, as to decrease the exposition time of the sample to the mechanical tensile stress (Fig. 5).

Comparing the strain values at $x = 0.8$ of these two experiments, we realize that the strain ε observed in Fig. 5 is mainly due to the expansion of the lattice ε_l. The obtained fit in function of stoichiometry is plotted in Fig. 5 (red line):

$$\varepsilon_l(x) = 1.61 \cdot x - 12.25 \cdot x^2 + 44.95 \cdot x^3 - 62.41 \cdot x^4 + 30.93 \cdot x^5. \quad (5)$$
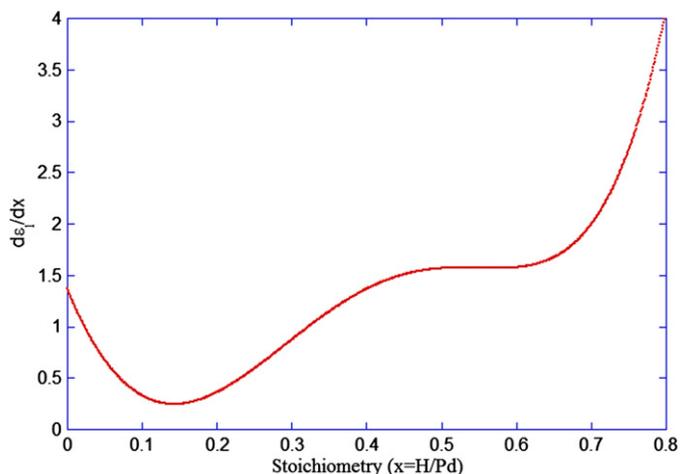
Fig. 6. Derivate of the fitted curve of Fig. 5 versus stoichiometry.

Fig. 7. Strain due to the mechanical tensile stress for "as drawn" and "annealed" samples as a function of H stoichiometry for a series of hydrogenation loadings (cathodic 10 V) on the same Pd sample (maximum error on ε_s: ± 0.02).

Fig. 8. Derivate of the curves of Fig. 7 versus stoichiometry.
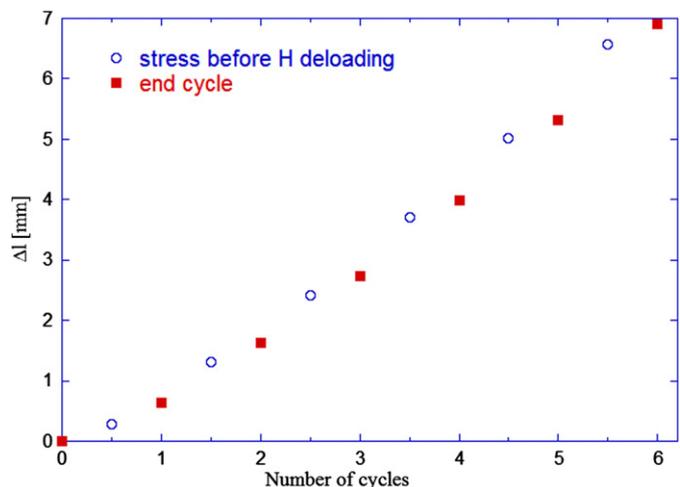
Fig. 9. Values of the residual length at the end of each hydrogenation (cathodic 10 V)/dehydrogenation (anodic 3 V) cycle (squares) and the extrapolated length due to ε_s before each H deloading (circles).

According to these data, the H adsorption causes an increase of the Pd lattice constant, a, of $1.036 \pm 0.008\%$ when going from a stoichiometry $x = 0$ to $x = 0.80 \pm 0.01$. Using the lattice constant $a = 3.89$ Å [3] for pure Pd, at a concentration of $PdH_{0.8}$ corresponds $a = 3.93$ Å. This value is in disagreement with the literature data available [2,10,16–20] which show for the stoichiometry $x = 0.8$ an increase of the lattice constant of about 3.5–4%, while our data is comparable with some recent work: in 2000 Numata et al. [23] report a relative elongation of about 1.7% at $PdH_{0.8}$; In 1999 Tsukuda et al. [22], observed an elongation of 0.16% at $PdH_{0.35}$ in accordance to our calculation. The differential variation of the Pd lattice constant in relation to H stoichiometry is obtained by calculating the ratio $\frac{d\varepsilon_l}{dx}|_{x=x*}$ from the fitted curve shown in Fig. 5. In which way H stoichiometry affects the increase of the Pd lattice constant for a given value x^* is shown in Fig. 6. A minimum value of $d\varepsilon_l/dx$ at stoichiometry of $x = 0.13$ is observed. For this stoichiometry value the insertion of H causes a very low modification of the lattice constant. Whereas at $x > 0.13$ the ratio $d\varepsilon_l/dx$ is an increasing function of the H content. In the region between $PdH_{0.6}$ and $PdH_{0.8}$, $d\varepsilon_l/dx$ increases exponentially, meaning that a little increase of H stoichiometry causes a huge increase of the lattice constant. As previously described, for each hydrogenation cycle, it's possible to evaluate ε_s, correlating the total percentage elongation ε with the corresponding ε_l reported in the same plot (red curves of Figs. 4 and 5), according to the relation (5). The ε_s as a function of the H concentration increases after each cycle (Fig. 7), and

particularly from about 0.4% (first cycle at $x = 0.7$) to about 1.6% (seventh cycle at $x = 0.7$).

Considering the differential of this function, for the first cycle a localized stress in the region $0.25 < x < 0.7$ is observed. For the other cycles the differential increases cycle after cycle, together with an extension of the region interested by the mechanical tensile strain ε_s. For each curve a maximum value of $d\varepsilon_s/dx$ at about $PdH_{0.52}$ is observed (Fig. 8). This means that a maximum effect of the H insertion on the strain due to the mechanical tensile stress is obtained at $x = 0.52$. This confirms that the residual strain at the end of each cycle is due to a combined effect of tensile stress and H dynamics [5] during H loading. By extrapolating the length increase only due to the mechanical tensile stress after H insertion inside Pd, the plot in Fig. 9 has been obtained. In this figure the values of the residual length at the end of each hydrogenation/dehydrogenation cycle (squares) and the extrapolated length due to ε_s before each H deloading (circles) are shown. After the first hydrogenation/dehydrogenation cycle, the contribution of the mechanical tensile strain due to the H loading is higher in respect to the one due to H deloading. In order to investigate if the metallurgical properties of the Pd wire influence the expansion of the lattice by hydrogenation, we performed the same experiments on annealed Pd samples as described in the introduction. The annealing procedure strongly affects the metallurgical state of
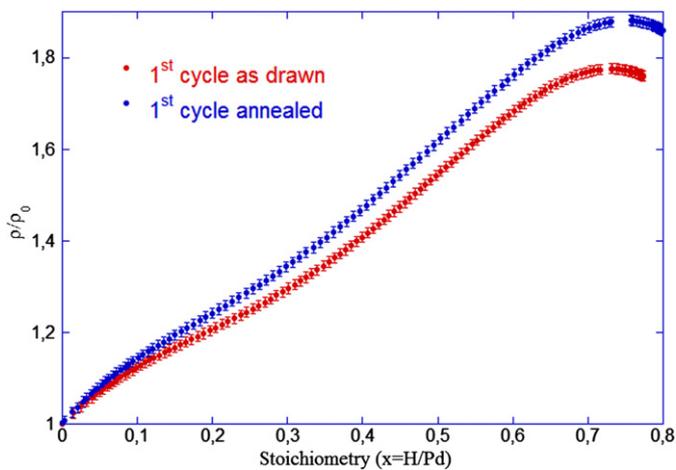
Fig. 10. Normalized resistivity as a function of H stoichiometry for first hydrogenation cycle (cathodic 10 V) of "as-drawn" and "annealed" samples.

Fig. 11. Derivate of the curves of Fig. 10 versus stoichiometry.

the sample, by changing the grains size, its average distribution and by decreasing the strength of the wire releasing stress. We performed again an experiment with 10 V, 50 V and 100 V potentiostatic electrolysis applying no mechanical tensile stress and measuring the length of the Pd wire only before ($x = 0$) and at the end ($x = 0.8$) of the hydrogenation cycle. Only the contribution of the strain due to the lattice expansion ε_l shows an elongation values comparable to "as drawn" sample values. This means that the metallurgical state of the metal does not influence the effect of the hydrogenation on the modification of the lattice constant. In Fig. 4, the relative elongation ε as a function of H content for three hydrogenation cycles of a Pd annealed stretched sample ($T = 0.49$ N) is plotted. As observed for the "as drawn" Pd wire, the strain ε is an increasing monotone function of the H content. On the contrary, a large increase of ε_s is observed with respect to the "as drawn" sample (Fig. 7), because of the Pd strength decrease caused by the annealing procedure. An unexpected decrease of ε_s is observed after the first H loading, in particular from about 3.2% (first cycle at $x = 0.7$) to about 1.6% (third cycle at $x = 0.7$). So, as previously reported [5], it has been confirmed that the metallurgical treatments only influence the mechanical characteristics of the Pd wire, do not affect the variation of the lattice constant due to H insertion, and that the mechanical strain is strictly connected to the variation of the electrical resistivity. The normalized electrical resistivity of the Pd sample versus its H content is not well documented in the literature. During the hydrogenation of a Pd wire, its electrical resistance is a function of the dimensional properties of the sample (length l and cross section S) and of the electron scattering centers (resistivity ρ):

$$R_{\mathrm{PdH}_x} = \rho_{\mathrm{PdH}_x} \cdot \frac{l_{\mathrm{PdH}_x}}{S_{\mathrm{PdH}_x}}. \qquad (6)$$

In our work the cross section can be determined from ε_l and ε_s by using (3) and (4). The initial cross section of the Pd wire is $S_o = (19.0 \pm 0.1) \times 10^{-6}$ cm^2, obtained by the SEM analysis (Jeol-JSM-6320F). From the length and the resistance measurements, together with the determined cross section, it is possible to calculate the electrical resistivity as a function of the H concentration inside the host lattice. The normalized resistivity in relation to the H content for the first H loading of both "annealed" and "as drawn" samples are plotted in Fig. 10. The annealed samples show higher values of normalized resistivity in respect to the "as-drawn" Pd, because the strain due to the mechanical tensile stress is almost double. In particular at stoichiometry $x = 0.73$, a difference of normalized resistivity of 5.6% is observed. At the same stoichiometry the strain due to mechanical tensile stress ε_s referred to the
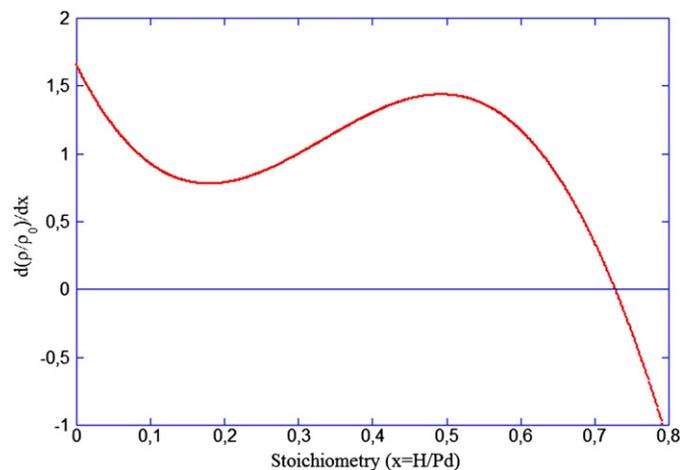
annealed sample is much higher (2.5%) in respect to the one observed for the "as drawn" Pd wire. The authors [5] have already shown that at the end of each hydrogenation/dehydrogenation cycle there is an electrical resistivity increase proportional to the residual strain, which can be explained with the Matthiessen's rule. Considering the derivative of the resistivity as stoichiometry function (Fig. 11), some analogies with the ε_l derivative (Fig. 6) are observed: at $x < 0.5$ the H insertion influences the expansion of the lattice and the creation of new scattering centers at the same rate; At $x > 0.5$ a large increase of $d\varepsilon_l/dx$ corresponds to the decrease of the resistivity. There is no theoretical explanation of this phenomenon in the literature.

3. Conclusion

The effect of H content on the relative elongation and on the electrical resistivity of a stretched Pd sample has been investigated for several hydrogenation cycles in a continuous range of H stoichiometry [$0 \leqslant x \leqslant 0.8$]. An increase of the electrical resistance at concentration $x = 0.73$ is observed cycle after cycle (30% after seven cycles), as observed in previous studies for deloaded Pd.

The effect of the Pd lattice expansion was found to be an increasing monotone function of the H content up to a strain of about 1.04% at the maximum concentration investigated. According to this, the lattice constant increases from $a = 3.89$ Å for pure Pd to $a = 3.93$ Å for PdH$_{0.8}$. This lattice expansion is in agreement with some recent work, but contradicts the known literature that claims about 4%. The effect of H insertion on the lattice constant variation has a minimum at $x = 0.13$ ($\alpha + \beta$ phase), and an exponential increase from $x = 0.6$ (β phase). From the total relative elongation of the wire, the contribution of mechanical tension applied has been separated from the lattice expansion previously mentioned. Cycle after cycle, an increase of the mechanical strain from 0.4% up to 1.6% at $x = 0.7$ is observed for "as drawn" samples, confirming a internal stress progressive increase of the Pd wire. Moreover, the effect of the H insertion on the increase of the mechanical strain shows a maximum contribution at PdH$_{0.52}$. We can conclude that H dynamics speeds-up the process of mechanical strain and increases the entity of this overall phenomenon during the H loading. A large increase of mechanical stress (3.2% at $x = 0.7$) is observed in annealed samples, while the same values of lattice constant expansion has been reported in respect to the "as drawn" samples. So, we can conclude that the metallurgical treatments only influence the mechanical properties of Pd wires, and not affect the variation of the lattice constant due to the H insertion. Some analogies between normalized resistivity and lattice expansion in relation to H content were found: for $x < 0.5$, the H

insertion influences the expansion of the lattice and the creation of new scattering centers at the same rate. At higher concentrations, a large increase of lattice expansion corresponds to the resistivity decrease. The annealed samples show higher values of normalized resistivity (up to 5.6% at $x = 0.73$) in respect to the "as drawn" Pd, because of a large difference of strain due to the mechanical tensile stress.

Acknowledgements

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