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KINETICS OF HYDROGEN PENETRATION INTO A CANTILE MADE OF HIGH-PURITY PALLADIUM AND α-PdH ALLOYS

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The influence of the rate of hydrogen supply into the chamber on the deformation process of a cantilever made of pure palladium and an α-PdH_n alloy at a temperature of 200 °C was studied. It was shown that the process of hydrogen saturation of palladium occurs in two stages: rapid bending, reaching the maximum bending with the formation of a plateau, and gradual straightening of the cantilever to a stationary state. It was experimentally established that the rate of hydrogen supply into the chamber affects the kinetics of the deformation process of a cantilever made of pure palladium, and is described by logarithmic functions, the duration of the plateau decreases with an increase in the rate of reaching the maximum bending.

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INTRODUCTION

With the rapid advancement of modern technologies in microelectronics, catalysis, energy storage, and precision instrumentation, the development and study of high-purity materials have become increasingly important [1–3]. Among the most widely used high-purity metals are palladium, platinum, copper, and silicon, which serve as key components in catalytic converters, hydrogen energy systems, and microelectronic devices [4–6]. For example, high-purity palladium is employed in hydrogen separation membranes, electrodes, and sensors due to its exceptional permeability to hydrogen and chemical stability [7, 8]. However, even minimal impurities or structural defects can alter the diffusion kinetics and mechanical behavior of such materials under operating conditions [9–11]. Quite often, bilayer samples are studied, where a palladium nanolayer is applied to a silicon cantilever sample and the bending of these cantilevers and the processes of hydrogen penetration into the palladium film are investigated [12–14]. In this kind of experiments, hydrogen diffusion in palladium is not considered because of the transience of this process compared to the process of hydrogen penetration into palladium, given the small thickness of the palladium film (nanolayer) [12–16]. Understanding these processes is essential for designing materials with predictable performance and long-term stability.

In this paper, we propose and carry out an experiment to measure the bending-extension of a relatively thick cantilever made of pure palladium in a hydrogen-filled chamber; we perform an asymptotic analysis and present the time dependence of the deflection of the palladium cantilever.

METHODS AND APPARATUS

An advanced hydrogen-vacuum unit (HVU) was used for the experiments [10, 17]. The samples, made in the form of cantilevers (a plate with dimensions of 68×5.5×0.27 mm made of palladium with a purity of 99.9%), were coated with a 750 nm thick layer of copper, which is impervious to hydrogen, on one side using an electrolytic method [18, 19]. After that, the samples were heated in the HWU chamber to the experimental temperature (273+200 K) and kept for 30 min to reduce thermal stresses. Then, hydrogen saturation of pure palladium cantilevers was carried out to the state of α-PdHn alloys, where n is a certain concentration of hydrogen in the alloy at a constant hydrogen pressure in the installation chamber. The resulting alloy was kept under isothermal conditions to relieve concentration stresses, after which additional one-sided saturation of the cantilever to the α-PdHn alloy was carried out, where n varies for each alloy by the same value. The process of changing the shape of the cantilever was recorded using a catheter and recorded on a video camera. The results were then interpreted and time dependencies of the cantilever shape change during the saturation process were plotted.

EXPERIMENT

A series of experiments was performed for a cantilever made of α-PdHn alloy, where ni+1=ni + Δn varies for each alloy depending on the hydrogen pressure in the chamber (Table 1). Table 1 shows the data on the change in the bending parameters of the palladium plate during the experiments with hydrogen penetration at different pressures, where P, MPa, is the hydrogen pressure during the injection; Δt_s, s, is the time of hydrogen injection into the chamber of the installation; Δt_{max}, s – time to reach the maximum bending of the cantilever; Y_{max}, mm – maximum bending of the cantilever; tp, s – duration of the plateau; Δt_{st}, s – time to reach the stationary state (residual bending) of the cantilever; Y_{st}, mm, is the value of the residual bending of the cantilever after relaxation; Δt_{ad}, s, is the duration of additional exposure of the cantilever in the hydrogen environment when the steady state does not change; n, H/Pd, is the ratio of the number of hydrogen atoms to palladium atoms (hydrogen concentration); Δn, H/Pd, is the addition of hydrogen concentration for the alloy; v, MPa/s, is the rate of hydrogen injection to a given pressure into the installation chamber. A series of experiments was conducted where the concentration varies by Δn = n, Δn = 1.6 n, and Δn = 1.4 n, respectively. Time dependencies of the change in the shape of the palladium cantilever with a change in the hydrogen supply rate were constructed (Figs. 1–3).

Table 1

Characteristics of cantilever shape changing at the rate of hydrogen supply to the chamber from $7.0 \cdot 10^{-3}$ to $16.6 \cdot 10^{-3}$ MPa/s

P, MPa	Δt_s, s	Δt_{max}, s	Y_{max}, mm	*tp*, s	Δt_{st}, s	Y_{st}, mm	Δt_{ad}, s	*n, H/Pd*	Δn, *H/Pd*	*v*, MPa/s
1. 0.0105	0.72	12	1.08	2	420	0.06	600	0.0053	0.0053	0.0146
2. 0.038	1.66	15	1.6	4	600	0.1	300	0.0138	0.00852	0.0166
3. 0.075	3.88	13	1.84	4	600	0.19	600	0.0214	0.0076	0.0067

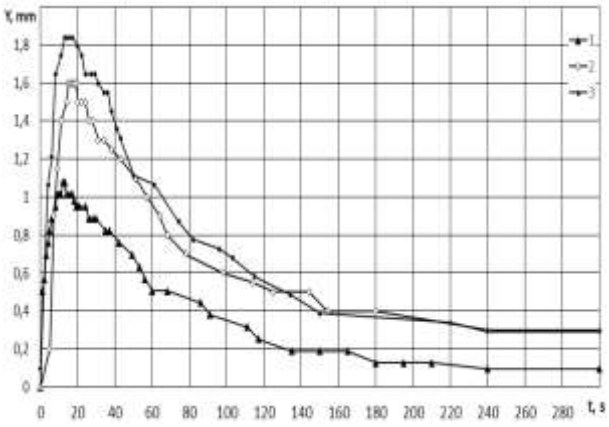

Fig. 1. Time dependences of cantilever shape change: 1 – for high-pure palladium; 2 – for α-$PdH_{0.005}$ alloy; 3 – for α-$PdH_{0.0138}$ alloy at a hydrogen injection rate from $7.0 \cdot 10^{-3}$ to $16.6 \cdot 10^{-3}$ MPa/s

Fig. 1 shows that the saturation process of cantilever from pure palladium and α-PdH_n alloy is carried out in two stages. For pure palladium (curve 1) and α-$PdH_{0.005}$ and α-$PdH_{0.0138}$ alloys (see Fig. 1, curves 2, 3), at the first stage, the cantilever bends rapidly immediately after the hydrogen is introduced into the chamber of the hydrogen-vacuum unit. At the same time, when the pressure gauge reaches the appropriate pressure (P) over time (Δt_s, s), which ensures the production of an alloy with the required concentration (n, H/Pd), the bending of the cantilever in all cases reaches an experimentally detectable value and continues to increase until it reaches the maximum value (Y_{max}, mm), and then a plateau with a duration of (tp, s) of several seconds is observed, and then the second longer stage of cantilever straightening begins with the achievement of a stationary state (Y_{st}, mm) over a certain time (Δt_{st}, s), which does not change during additional exposure (Δt_{ad}, s).

Let us consider the first experiment in more detail (see Fig. 1, curve 1), when the high-purity palladium cantilever with is saturated to form the alloy α-$PdH_{0.0053}$. Hydrogen is introduced into the chamber to a specified pressure of 0.01 MPa in 0.72 s at a rate of $14.58 \cdot 10^{-3}$ MPa/s, and the concentration changes accordingly to $\Delta n = 0.0053$ H/Pd. From the moment hydrogen begins to be pumped into the chamber, the cantilever deflection continues to increase and reaches its maximum value of $Y_{max} = 1.08$ mm in 12 s and stabilises in this position for 2 s. Then the cantilever begins to straighten and the deflection arrow decreases. This is the beginning of the second stage, which is almost 26 times longer than the first stage (see Fig. 1, curve 1). After 420 s from the start of the experiment, the cantilever reaches a steady state ($Y_{st} = 0.06$ mm), which does not change during the subsequent long exposure of 600 s.

In the second part of this experiment, if the pressure in the chamber is increased from 0.010 to 0.038 MPa over $\Delta\tau_s = 1.66$ s, an α-$PdH_{0.0138}$ alloy is obtained with a change in concentration of $\Delta n = 0.0085$ H/Pd, which is greater than in the previous experiment, and a greater maximum cantilever deflection ($Y_{max} = 1.6$ mm) was experimentally recorded for $\Delta t_{max} = 15$ s (see curve 2 in Fig. 1) from the start of hydrogen injection into the chamber. Next, we observe a plateau lasting 4 s, after which the cantilever begins to straighten gradually, and this process slows down over time until a certain steady state ($Y_{st} = 0.1$ mm) is reached after 600 s. An additional exposure for 300 s did not lead to a change in the position of the cantilever.

In the third part of this experiment, the pressure in the chamber was increased from 0.038 to 0.075 MPa ($\Delta n = 0.0076$ H/Pd) over $\Delta\tau s = 3.88$ s, resulting in an alloy of α-$PdH_{0.0214}$. Experimentally, a greater maximum cantilever deflection ($Y_{max} = 1.84$ mm) was recorded for $\Delta\tau_{max} = 13$ s (see curve 3 in Fig. 1) from the start of hydrogen injection into the chamber and a plateau lasting 4 s, after which the cantilever began to straighten until it reached a certain steady state ($Y_{st} = 0.19$ mm) in 600 s. An additional exposure for 600 s did not change the position of the cantilever.

The second experiment was conducted with a reduction in the hydrogen supply rate by almost 20 times (Table 2 and Fig. 2).

From Fig. 2 we see that when the rate of hydrogen supply into the chamber changes by almost 20 times compared to experiment 1 at the first stage (see curve 1 in Fig. 2), the increase in the maximum bending when saturated with pure palladium occurs proportionally to the time of hydrogen supply into the chamber (for 19 s), however, after the hydrogen supply into the chamber is stopped, the value of the maximum bending stabilizes and a plateau is formed with a duration of 7 s.

Table 2

Characteristics of the cantilever deformation when changing the hydrogen supply rate into the chamber from $3.2 \cdot 10^{-4}$ to $5.4 \cdot 10^{-4}$ MPa/s

P, MPa	Δt_s, s	Δt_{max}, s	Y_{max}, mm	*tp*, s	Δt_{st}, S	Y_{st}, mm	Δt_{ad}, s	*n*, H/Pd	Δn, H/Pd	$v \cdot 10^{-4}$, MPa/s
1. 0.0105	19.6	19	1.06	7	600	0.12	600	0.0053	0.0053	5.4
2. 0.038	59.9	62	1.3	7	1380	0.2	1000	0.0138	0.00852	4.6
3. 0.075	84.7	92	1.35	5	480	0.3	420	0.0214	0.0076	3.2

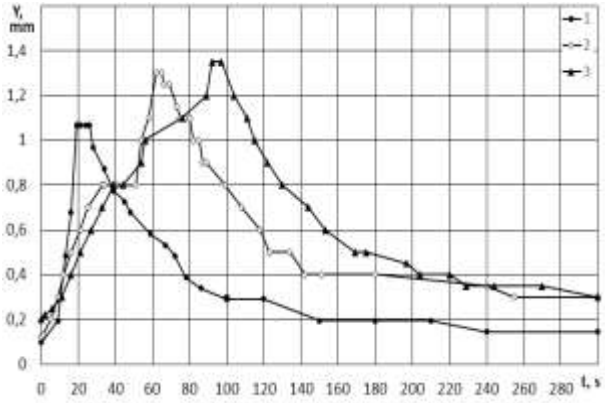

Fig. 2. Time dependence of cantilever deformation 1 – for high-pure palladium; 2 – for α-$PdH_{0.005}$ alloy; 3 – for α-$PdH_{0.0138}$ alloy when the hydrogen inlet rate into the chamber is reduced by almost 20 times

When the alloy is saturated with α-$PdH_{0.053}$ and α-$PdH_{0.0138}$, the maximum bend is achieved after the hydrogen supply to the chamber is stopped for 62 and 92 s, respectively (see Fig. 2, curves 2 and 3, respectively) and is accompanied by the formation of a plateau for 7 and 5 s, respectively. The second stage of shape change here is characterised by a smooth and slow straightening and reaching a steady state of 0.2 mm in 1380 s and does not change during an additional holding time of 1000 s (curve 2). For curve 3, the residual deflection is 0.3 mm after 480 s (see curve 3 in Fig. 3) from the start of hydrogen injection into the chamber and remains unchanged during an additional 420 s of exposure.

The third study was conducted with the same concentration changes as in the first and second experiments, but the hydrogen supply time was increased by 1.5 times compared to the second experiment (see Fig. 3 and Table 3).

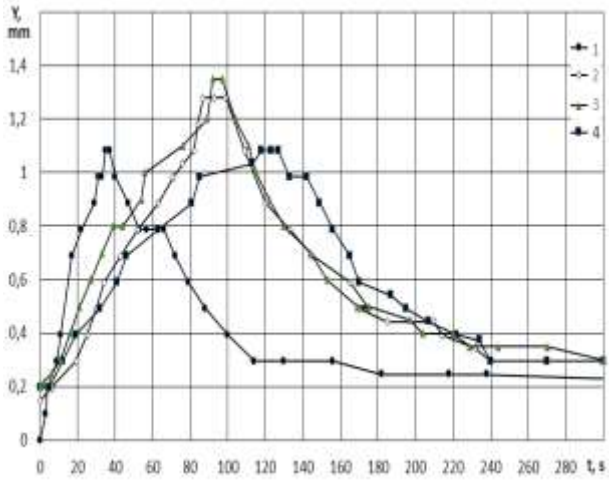

Fig. 3. Time dependences of cantilever shape change: 1 – for high-pure palladium; 2 – for α-$PdH_{0.0053}$ alloy; 3 – for α-$PdH_{0.0138}$ alloy at a hydrogen injection rate of into the chamber of $3.2 \cdot 10^{-4}$ MPa/s; 4 – for the α-$PdH_{0.0138}$ alloy at a hydrogen inlet rate into the chamber of $2.2 \cdot 10^{-4}$ MPa/s

Table 3

Characteristics of the cantilever deformation when changing the hydrogen supply rate into the chamber from $3.2 \cdot 10^{-4}$ to $3.4 \cdot 10^{-4}$ MPa/s

P, MPa	Δt_s, s	Δt_{max}, s	Y_{max}, mm	*tp*, s	Δt_{st}, S	Y_{st}, mm	Δt_{ad}, s	*n*, H/Pd	Δn, H/Pd	$v \cdot 10^{-4}$, MPa/s
1. 0.0105	31.09	31	1.08	9	655	0.15	600	0.0053	0.0053	3.4
2. 0.038	84.06	87	1.27	12	480	0.19	520	0.0138	0.00852	3.3
3. 0.075	85	93	1.36	5	500	0.3	500	0.0214	0.0076	3.2
4. 0.075	122.9	118	1.08	9	900	0.22	600	0.0214	0.0076	2.2

As we can see from Table 3, all three experiments were conducted at almost the same speed of $3.2 \cdot 10^{-4}$ MPa/s, and as a result, the maximum deflection increases for 1 run to 1.08 mm in 31 s and remains at a plateau for 9 s, after which the plate straightens and returns to its steady state to 0.15 mm in 655 s, and during 600 s of additional exposure, the residual deformation remains unchanged. When the alloy is saturated with α-$PdH_{0.053}$ and α-$PdH_{0.0138}$, the maximum deflection is reached after the hydrogen supply to the chamber is stopped for 87 and 93 s (see Fig. 3, curves 2 and 3, respectively) and is accompanied by the

formation of a plateau for 12 and 5 s, respectively, followed by straightening (curve 2 and curve 3) so after 100 s from the start of the injection, the kinetics of the straightening of these two curves proceed similarly, because the injection of hydrogen into the chamber occurred at approximately the same rate. However, the magnitude of the residual deformation increases with increasing hydrogen content for the α-$PdH_{0.053}$ alloy and is 0.19 mm in 480 s and 0.3 mm for the α-$PdH_{0.0138}$ alloy in 500 s, and does not change during additional holding.

It is interesting to analyse another experiment in which the α-$PdH_{0.0138}$ alloy was saturated, but at a rate 1.5 times lower than for curve 3. Thus, on curve 4, even during the hydrogen injection process, the sample reaches a maximum deflection less than that for curve 3 (1.08 mm) and a plateau lasting 9 s is formed, and then, starting from 140 to 240 s, the cantilever straightens over 100 s, and as we can see, the kinetics of straightening for curves 2, 3, and 4 are similar. The value of the residual deformation at a lower feed rate also decreases over 900 s to 0.22 mm.

RESULTS AND DISCUSSION

Let us analyse the results obtained in Figs. 4–6, when changing the maximum deflection value in three experimental series (curves 1, 2, 3 describe the 1st, 2nd, and 3rd passes).

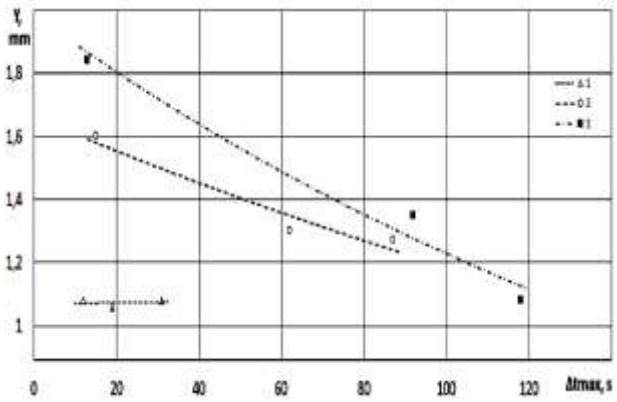

Fig. 4. Dependence of the maximum cantilever deflection on the time to reach maximum deflection:
1 – for high-pure palladium (overlay 1);
2 – for alloy α-$PdH_{0.0053}$ (overlay 2);
3 – for alloy α-$PdH_{0.0138}$ (overlay 3)

Thus, with increasing hydrogen content, the maximum bending value increases, with a change (increase) in the hydrogen supply rate, the time to reach the maximum increases. At a temperature of 200 °C for 1 pass, the shortest time range was obtained, a sharp drop in the maximum with an increase in the time to reach the maximum can be described by an exponential approximation in the form:

$$Y_{max} = 1.069e^{-0.0002\,t},\ R^2 = 0.022,$$

where t – is the time to reach the maximum; R^2 – is the coefficient of determination showing how well the model describes the data.

The second pass is characterized by a lower slope of decline than the first pass and an exponential approximation in the form

$$Y_{max} = 1.6615e^{-0.003\,t},\qquad R^2 = 0.9451,$$

The 3rd pass is characterized by the largest maximum value with a longer reach time, which decreases slowly. It is best approximated by an exponential:

$$Y_{max} = 1.968e^{-0.005\,t},\ R^2 = 0.973,$$

As we can see, with increasing time to reach maximum bending for 3 overlaps, the bending value demonstrates an exponential decrease in the maximum value with increasing time, i.e. the longer the system reaches the maximum, the smaller the maximum bending value.

In Fig. 5, the curves can be approximated by logarithmic dependences of the bending magnitude on the feed rate. For each pass, a logarithmic curve of the form is constructed:

For pass 1: $Y_{max} = 0.0022 \ln(v) + 1.0881$, $R_2 = 0.3112$;
For pass 2: $Y_{max} = 0.0839 \ln(v) + 1.9441$, $R_2 = 0.6193$;
For pass 3: $Y_{max} = 0.1963 \ln(v) + 2.8264$, $R_2 = 0.9397$,

where v is the hydrogen feed rate into the chamber, MPa/s.

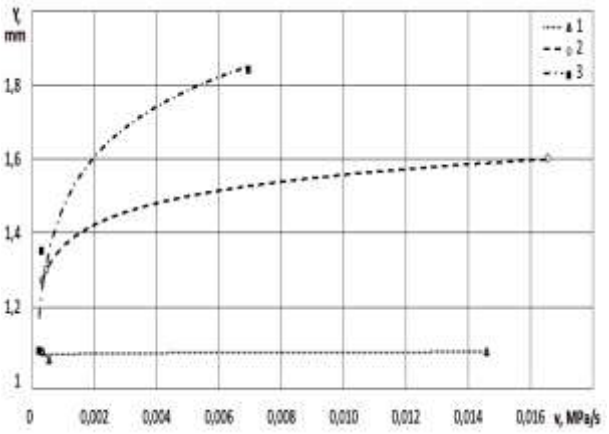

Fig. 5. Dependence of the maximum cantilever bending on the hydrogen supply rate into the chamber:
1 – for high-pure palladium (overlap 1);
2 – for the α-$PdH_{0.0053}$ alloy (overlap 2);
3 – for the α-$PdH_{0.0138}$ alloy (overlap 3)

An increase in the feed rate leads to an increase in bending, but the dependence is not linear – it is described by a logarithmic function. Thus, for 3, 2, 1 passes, an increase in the maximum bending is observed with increasing feed rate, which coincides with the experimentally obtained data.

At a temperature of 200 °C, it is noted that the higher the hydrogen concentration in the alloy, the higher the sensitivity of the material to the feed rate: α-$PdH_{0.0138}$ > α-$PdH_{0.0053}$ >> Pd.

Let us analyses the duration of the plateau for each pass from the rate of hydrogen supply to the chamber (see Fig. 6).

As we can see, when the hydrogen injection rate decreases, the plateau duration increases – a clear decline is observed for all three curves. Thus, for pure

palladium (curve 1), the plateau value is located between the α-$PdH_{0.0053}$ and α-$PdH_{0.0138}$ alloys and indicates a slightly slower hydrogen absorption process.

The high feed rate indicates intense interaction between hydrogen and palladium, the emergence of hydrogen concentration stresses, and an intense process of redistribution of these stresses at the moment of reaching the maximum bend, which is accompanied by a shorter plateau. Thus, at the beginning of the filling, the diffusion mode dominates, ensuring a high feed rate. Upon reaching a certain saturation level, kinetic limitations begin to take effect, significantly reducing the gas absorption rate.

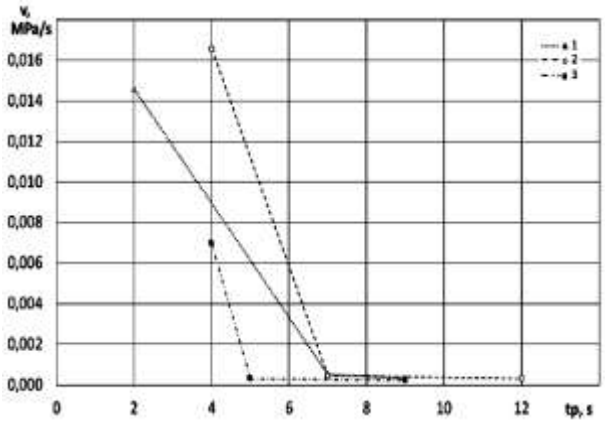

Fig. 6. Dependence of the plateau duration on the hydrogen supply rate into the chamber: 1 – for high-pure palladium (overlay 1); 2 – for alloy α-$PdH_{0.0053}$ (overlay 2); 3 – for alloy α-$PdH_{0.0138}$ (overlay 3)

As we can see from the experimental results, various factors influence the maximum deflection value. A decrease in the plateau with an increase in the speed of reaching the maximum is observed in all three infusions (curves 1–3):

– this indicates that quickly reaching the maximum deflection of a larger amplitude leads to a shorter plateau;

– at the lowest speed of reaching the maximum deflection, the time to reach the plateau increases, as does its duration;

– the higher the hydrogen content in the α-PdHn alloy, the longer and more intense the maximum deflection is reached;

– the third run has the highest starting values, but declines the fastest, which may indicate a changed material structure (e.g., higher palladium saturation with hydrogen in the $PdH_{0.013}$ alloy).

CONCLUSIONS

During a series of experiments, a comparative analysis was conducted of the deformation response of cantilevers made of high-purity palladium (batch 1) and palladium alloys with varying degrees of hydrogenation: α-$PdH_{0.0053}$ (batch 2) and α-$PdH_{0.0138}$ (batch 3). The main focus was on studying the dependencies of the maximum deflection, the time required to reach it, the hydrogen feed rate, and the plateau parameters at saturation.

1. The dependence of the maximum deflection on the time to reach the maximum was obtained for all three samples, where an exponential decrease in the maximum deflection was observed with an increase in the time required to reach it. This indicates the kinetic limitation of the deformation process: a faster reaction is accompanied by a more intense deflection.

2. It was established that the maximum deflection of a cantilever made of pure palladium and α-PdHn alloy depends on the hydrogen supply rate and is described by logarithmic functions of maximum growth with increasing supply rate (more intense hydrogen supply causes a stronger reaction).

3. For the first time, it has been experimentally obtained that when saturating α-PdHn alloys with a change in hydrogen concentration in the alloy by the same amount and at the same rate, the maximum deflection of the cantilever is constant.

4. It has been established that the duration of the plateau decreases with an increase in the rate of reaching the maximum deflection. Therefore, the plateau value is inversely dependent on the speed of reaching the maximum – a typical indicator that a fast process does not have time to stabilise, while a slow process forms a longer plateau.

5. The data confirm the promise of using palladium alloys in sensor and diagnostic systems for hydrogen monitoring. An increase in the hydrogen content in the α-PdHn alloy leads to a longer plateau with a slower increase in hydrogen concentration, which may be useful for the development of hydrogen sensors with a delayed response.

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КІНЕТИКА ПРОНИКНЕННЯ ВОДНЮ В КАНТИЛЕВЕР ІЗ ВИСОКОЧИСТОГО ПАЛАДІЮ ТА ЗІ СПЛАВІВ α-PdH

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Проведено дослідження впливу швидкості подачі водню в камеру на процес формозмінення кантилевера чистого паладію і сплаву α-PdH_n за температури 200 °С. Показано, що процес насичення паладію воднем відбувається у два етапи: швидке згинання, досягнення максимального вигину з утворенням плато та поступове розпрямлення кантилевера до стаціонарного стану. Експериментально встановлено, що швидкість подачі водню в камеру впливає на кінетику процесу формозмінення кантилевера з чистого паладію та описується логарифмічними функціями, тривалість плато зменшується зі збільшенням швидкості досягнення максимального вигину.